

State of Illinois  
DEPARTMENT OF TRANSPORTATION  
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FIELD EVALUATION OF MICROWAVE PATCHING SYSTEM

by

R. J. Little

Final Report

Research Study IHR-902

A Research Project Conducted by  
Illinois Department of Transportation  
in cooperation with  
U. S. Department of Transportation  
Federal Highway Administration

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## FIELD EVALUATION OF MICROWAVE PATCHING SYSTEM

### INTRODUCTION

The concept of combining microwave power and polymer concrete to obtain a rapid curing material for road and bridge patching was developed by Syracuse University Research Corporation, presently known as Syracuse Research Corporation (SRC). Through a research program sponsored by the Federal Highway Administration and five states, Connecticut, Illinois, Maryland, Ohio, and Pennsylvania, SRC developed a Microwave Heating System for road maintenance. This system, delivered to Illinois in April 1977 for field evaluation, is the subject of this report.

Polymers have been in use for many years. Only recently have they been introduced into highway technology for pavement and bridge maintenance. Probably the most familiar polymerization product is that of the epoxy resin glues, which have been used in the construction industry for some time, and more recently in the highway field.

In general, the polymerization reaction occurs when a monomer molecule is provided with the proper conditions to react with other monomers. A monomer is a chemical compound that can undergo polymerization. This is most familiar with the epoxy glues when the resin and hardener are mixed in a predetermined quantity, which causes a chemical reaction creating the polymerization process.

Most monomers, when combined, react to form a polymer at room temperature. Usually the reaction is inhibited by low temperatures and accelerated by high temperatures. The reaction is exothermic and this tends to provide additional heat to speed up the process. A complete

discussion of polymer chemistry relative to selecting a satisfactory product for highway maintenance is covered in detail in Final Reports SURC TR 73-436 (1), and SURC TR 76-052 (2).

The use of microwave energy has been employed in a variety of ways. However, this is the first time microwave energy has been used in highway technology as a maintenance tool. The unit used in this project, developed by SRC, is discussed fully in the same reports previously mentioned (1), (2).

Further discussion of the polymers and the microwave equipment will be only relative to this study and the experience gained by working with them. This report will cover the chemicals and catalyst used in the resin system, the microwave-generating equipment, aggregates, mixes, field experience, laboratory work, and safety.

#### Study Objectives

The primary objective of this study was to evaluate the microwave heating process for repairing concrete pavements and bridge decks under operating field conditions using regular maintenance field crews. The microwave technique was to be evaluated as a process for performing the following specific tasks.

- (1) Full- and partial-depth patching of bridge decks
- (2) Full- and partial-depth CRC pavement patches
- (3) Patching of joints on jointed PCC pavements

A secondary objective of the study was to include the testing and evaluation of different aggregates for patching with the system.

#### Background

The term microwave defines a range of the electromagnetic spectrum which lies between radio waves and infrared light. As the microwaves

are allowed to pass through a material containing molecules having a pair of closely spaced electric charges, equal in magnitude but opposite in sign (dipole molecules), the microwaves create an energy field in the material which causes reorientation of the charged molecule to align with the field of energy. The microwave unit developed for concrete patching produces waves with a frequency of 2,450 Mega Hertz (2.45 billion cycles per second). With this frequency, the change in orientation of the charged molecules occurs at a rate of 2.45 billion times per second, creating considerable molecular friction and a subsequent heat rise.

Generation of heat in the material subjected to microwave energy is only possible when the material is of a type which absorbs such energy. Material which reflects the major portion of incident energy or is transparent (i.e., permits energy to pass through without appreciable loss) is not suitable for microwave heating.

Penetration by the microwave energy through an absorbing material is not uniform, with the energy available for heating being diminished as it progresses through the material. This results in non-uniform heating of the surface, with the larger portion of heat being dissipated closer to the surface. For this reason, a material with some transparency is needed to allow adequate penetration of the microwave energy for proper curing throughout the depth of the material.

During the development of the microwave patching process, many materials and combinations of materials were evaluated by SRC for use in concrete patching. The most successful material tested by SRC to date is a polyester resin in solution with styrene which, under heat, bonds the polyester molecules together to form a unified mass.



The polyester resin by itself is transparent; therefore, it cannot be evaluated alone. The aggregate in the mix is the material that absorbs the microwave energy. The aggregate should not be oven dry, but only surface dry, since the lack of water in an oven-dry aggregate diminishes the capability of the patch material to convert sufficient microwave energy to thermal energy.

Two essential properties are required for chemical systems used in pavement repair applications. These systems must have:

- (1) Sufficient internal physical strength to yield a material that will not fail in field use
- (2) Sufficient adhesion to hold the patch to the surrounding sound material

The polyester resin tested by SRC appears to meet all of the physical and chemical criteria required for successful patching of concrete pavements. The work of this study dealt primarily with the materials recommended by SRC.

#### Equipment

The microwave applicator, developed by SRC, and its support equipment were designed to fit on a truck supplied by the Illinois Department of Transportation. The total mobile system includes the truck, a 50 kw capacity diesel generator, a 20 kw microwave power generator (applicator), a cooling system, and a hydraulic crane. The system was well conceived, and functions well for highway maintenance. There were some problems with the equipment that will be discussed later. The system is seen in Figure 1.

The microwave applicator is made up of eight, 2.5 kw, magnetrons in the microwave power generating unit which supplies a total of 20 kw

of power output. A control panel on the applicator provides for individual unit setting. Each unit radiates a given area of the total area covered by the applicator. The inside dimensions of the applicator are 29 inches (0.74 m) by 43 inches (1.09 m), which means the nominal area that can be cured at one setup is 8.5 square feet (0.8 sq. m). Each unit has an on and off switch and either low or high power mode for radiating. These features provide for a wide range of operating conditions when curing a patch. A complete operating procedure is given in Appendix A.

#### Resin System

The chemicals recommended by SRC for this system are polyester, styrene, and a catalyst or initiator. Two essential properties required for the chemical system are: (1) sufficient internal physical strength to yield a material that will not fail in field use, and (2) sufficient adhesion to hold the patch to the surrounding "sound" material.

The requirements set for the resin system are that it should conform to the following:

- (a) Unsaturated thermosetting polyester resin
- (b) Low-to-medium exotherm with low shrinkage
- (c) Viscosity in the 700 to 1000 cps range
- (d) Polymerization not unduly affected by air or moisture
- (e) Unpromoted resin

Chemicals meeting the above requirements are readily available from several major manufacturers.

The catalyst recommended for this resin system in order of preferred use are: (1) Vazo-64, (2) Methyl Ethyl Ketone Peroxide, and

(3) Benzoyl Peroxide. The Vazo-64 was selected because it is somewhat more temperature selective and is less reactive at room temperature. It also causes a faster rate of polymerization at temperatures lower than the peroxide catalyst.

The resin system was recommended to be 60 percent polyester and 40 percent styrene. The recommended amount of catalyst was 2 percent by weight of the resin when using Vazo-64 and one percent when using either of the peroxides. The amount of resin recommended was 10 percent by weight of the aggregate.

#### Aggregate

The recommended aggregate was a blend of coarse aggregate (crushed stone), fine aggregate (natural sand), and kiln dust. The typical gradation recommended was as follows:

<u>Sieve Size</u>	<u>Percent Passing</u>
1/2 in.	100
1/4 in.	68
1/8 in.	46
No. 10	30
No. 20	23
No. 40	17
No. 80	8
No. 200	6

The kiln dust was shown to increase the strength of the mix. However, cement or mineral filler may be substituted for the kiln dust with good results. Cement was used for all patches because of its availability.

While the use of kiln dust was recommended by SRC, they suggested that either cement or mineral filler could be used as a substitute for the kiln dust. Both kiln dust and mineral filler are less expensive than cement. However, cement was the most readily available and was therefore used. When patching on a larger volume the acquisition of less expensive

materials should be considered. For further effects of the use of kiln dust in the mix the reader is referred to Final Report SURC TR-73-436(1).

#### POLYMER CONCRETE

Polymer concrete is a blend of aggregate and cementing agent. In this case the cementing agent is the resin system which takes the place of portland cement and water in portland cement concrete.

Because this study primarily was a field evaluation, laboratory work was kept to a minimum. However, a certain amount of laboratory work was necessary to acquaint the observer with the characteristics of polymer concrete and design the mix for the different aggregates to be used, which satisfies the secondary objective.

#### Aggregate - Mix Design

When it had been determined that the field study would be conducted in Highway Districts 3, 4, and 6, they were requested to submit samples of the aggregate they normally use for making small conventional PCC patches. Districts 3, 4, and 6 headquarters are located in Ottawa, Peoria, and Springfield, respectively.

Districts 4 and 6 submitted pea gravel and District 3 submitted a crushed limestone, all with similar gradation. All three districts submitted sand of similar gradation.

In determining the proportions of aggregate to be used in the trial mix for each district, two criteria were kept in mind, first, to come as close as possible to duplicating the recommended mix and, second, arrive at a combination of coarse and fine aggregates that would be easy to measure in the field. The gradations established for each district are in Table I.

TABLE 1. AGGREGATE GRADATION PERCENT PASSING

<u>Sieve Size</u>	<u>District 3</u>	<u>District 4</u>	<u>District 6</u>
1/2	100	100	100
3/8	86.3	99.8	97.3
4	43.7	50.1	58.0
10	23.8	30.3	31.3
16	18.7	24.2	26.9
20	15.5	18.4	23.3
40	5.6	4.2	9.6
80	0.6	1.1	0.9
200	0.3	0.3	0.2

To obtain these gradations in the field the weight of each aggregate per patch was determined as follows:

	<u>District 3</u>	<u>District 4</u>	<u>District 6</u>
Coarse Aggregate	122 lbs.	128 lbs.	125 lbs.
Field Aggregate (Sand)	61 & 74 Lbs.	70 lbs.	70 lbs.

The two values for sand in District 3 represent a field change after the first few patches were made.

The fine material, cement, was set at 18 lbs (8.2 kg) per batch for all districts. To facilitate proportioning in the field and to eliminate the need for scales, five-gallon pails were marked to obtain the correct weight by volume.

Water is a very important ingredient necessary for microwave energy to raise the temperature of the mix to obtain polymerization. The aggregate should contain some moisture but have a dry surface. Since this was the condition of the aggregate samples, no correction was made for moisture.

#### Chemicals

The chemicals as recommended were used. Initially, the recommended proportions of polyester, styrene, and catalyst were used. Some changes were made after consultation with SRC and field experience indicated the need for adjustment of the resin system. Two different proportions of resins were used and two different amounts of catalyst were used. The amount of resin used per batch was varied several times. Charts used to proportion the two different amounts of resin using 1.5 percent catalyst are shown in Figures A-1 and A-2 of Appendix A.

### Acquisition, Storage, and Handling

Several manufacturers make the chemicals required for this system. The obtainability of the chemicals in order from easiest to most difficult to obtain is the styrene, polyester, and catalyst. The developer of the system, SRC, investigated possible sources and found that most major manufacturers of unsaturated polyester resins categorized resins with the properties required for this system.

To obtain the chemicals required, several companies were contacted. Due to constraints on the quantities required for this study, and order limits by some of the manufacturers, acquisition was limited to certain suppliers. In addition to the chemicals required for the mix, a solvent was required for cleaning tools and the mixer. The solvent used was methylene chloride.

The liquid chemicals were received in 55-gallon (208.2-l) barrels. The weights of the barrels are about 500 lbs. (226.8 kg), 400 lbs. (181.4 kg), and 600 lbs. (272.2kg) each for the polyester, styrene, and methylene chloride, respectively. The weight of full barrels is sufficient to require mechanical assistance in handling. A suitable tool for handling the barrels is a modified hand truck that allows the barrels to be changed from the upright position to a horizontal position for draining the barrels.

Storage of the chemicals is critical. The manufacturer suggested a shelf life of six months for the polyester. The shelf life for this chemical can be extended to several times that length by keeping it in a cool place.

The styrene is more sensitive to storage conditions than the polyester, and should be inspected frequently. At first the polyester and

styrene were stored in a building with adequate ventilation until the material in the bottom of the styrene started to get thick. At that time the polyester and styrene were moved into a small room with air conditioning. When the air conditioner broke, the room became very warm and the styrene polymerized. This problem pinpoints the need for regular inspection of the chemicals and storage conditions.

The catalyst used was Vazo-64. It thermally decomposes to form free radicals, which initiates a polymerization reaction and is therefore unstable at higher temperatures. The catalyst should be kept in a cool, dry environment. A refrigerator was obtained for use as a storage cabinet for the catalyst.

Orders for any of the chemicals should be made when ambient air temperatures are most conducive to that chemical. Since all the chemicals are best preserved in a cool environment, orders should be obtained from October to May. This eliminates a problem of shipping during hot weather when the chemicals could be affected by high temperatures. As an example, a quantity of Vazo-64 was delayed in shipping and sat on a loading dock for 3 weeks in late October. The temperature for that period was cool, so no problems arose. Three weeks on a loading dock in July, however, could have been a problem.

The converse is also important. Some chemicals will not survive a freeze. A one-gallon sample of a different catalyst was received in the winter in a frozen state. Upon thawing, this sample was totally useless.

Freezing of the polyester and styrene does not seem to affect their function. Intentional freezing of the two resulted in a very thick material not conducive to mixing with aggregate, but they still produced a quality mix.



## EQUIPMENT

The special equipment necessary for patching with this system is the Microwave Power Generating System and some safety equipment. Other equipment is that normally used when patching with portland cement concrete. This section covers training of the crew in using the system, safety procedures, safety equipment necessary, and crew reaction to the system.

### Training and Safety

When the unit was delivered by SRC in April 1977, a two-day training session was set up for key personnel from each district. The first-day session was a classroom-type introduction to the system, with a heavy emphasis on safety. The second day was devoted to actual use of the equipment and individual instructions for the men who would be operating the equipment in each district. Again, heavy emphasis was placed on safety.

Each district had a crew member assigned to operate the microwave system and, when available, one crew member was assigned the job of mixing the chemicals. The operators were experienced men and soon learned to operate the system. Because the system was designed to be operated in a sequential order, a copy of the sequence as listed in Appendix A was kept on the truck for reference. The operator also was instructed on how to use the hazard meter for detecting radiation leaks and how to correct them. The crew member in charge of mixing the chemicals was instructed on safety procedures to be followed when mixing the chemicals.

When the unit was moved into a district for the first time or when crew members changed, a short explanation of the system was given to the crew members. Safety measures were explained, and excellent cooperation was received. After the first day's operation, the crew members were asked about their feelings of working with such a system. Approximately 20 men worked one or more days with the system, and only one had a negative comment. He had a fear of the microwave and would not get close to it when radiating. The high degree of acceptance from the crews indicates that there should be no problem with worker acceptance of a system of this type for general use, especially after a full explanation of the system and safety practices required.

Safety equipment that is absolutely necessary is:

- Goggles
- Respirators
- Clean water
- Hazard meter
- First aid kit
- Portable oxygen supply
- Ear protectors
- Gloves

The number of goggles and respirators required may vary from crew to crew, depending on work assignments. However, anyone coming in contact with the chemicals should have them. Five sets usually are adequate. The clean water is for flushing the eyes in case of accidental splashes that may get in the eye. The hazard meter is for detecting radiation leaks around the microwave applicator while curing a patch in the radiation mode. The applicator is equipped with two rows of shielding on the

bottom which compresses when the applicator is set over a patch. However, small irregularities in the pavement can cause leaks of radiation. Once detected, sandbags should be placed at the source of the leak. Few leaks above the safe level were detected, and then a couple of sand bags were sufficient to reduce the leak to the safe level. The first aid kit is for the usual treatment of the common cuts and scrapes.

The portable oxygen supply is a first aid item rather than a preventative-type safety equipment. The fumes given off by the resins are not unpleasant to some people and they therefore tend to disregard the benefits of the respirators. However palatable the odor may be, the effects are the same if overexposure happens. Part of the effect is weakness and dizziness. One person finishing a patch refused to wear a respirator and, when he finished the patch, showed signs of weakness and dizziness. He was given ten minutes on the oxygen supply and was required to remain seated until the condition passed. This was the only safety problem encountered in the field during the study, and should be a reminder for the future that anyone working in the fumes for more than a few minutes should be required to wear a respirator. The respirators used had screw-on-type air filters with activated charcoal to eliminate fumes and dust. These respirators are available at many hardware stores along with the replacement filters, and are relatively inexpensive. Properly fitted, these filters eliminate all odors given off by the resins. The goggles also are readily available and protect the eyes from any splashing of the chemicals while handling and mixing. Pictures of the essential safety equipment are seen in Figures 2 and 3.

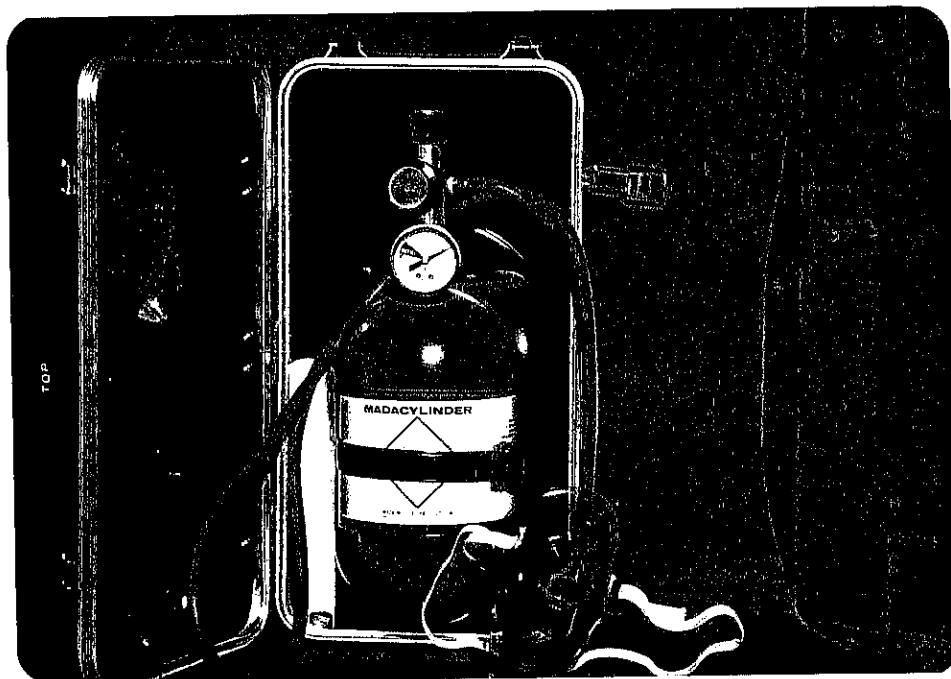


Figure 2. First aid oxygen supply

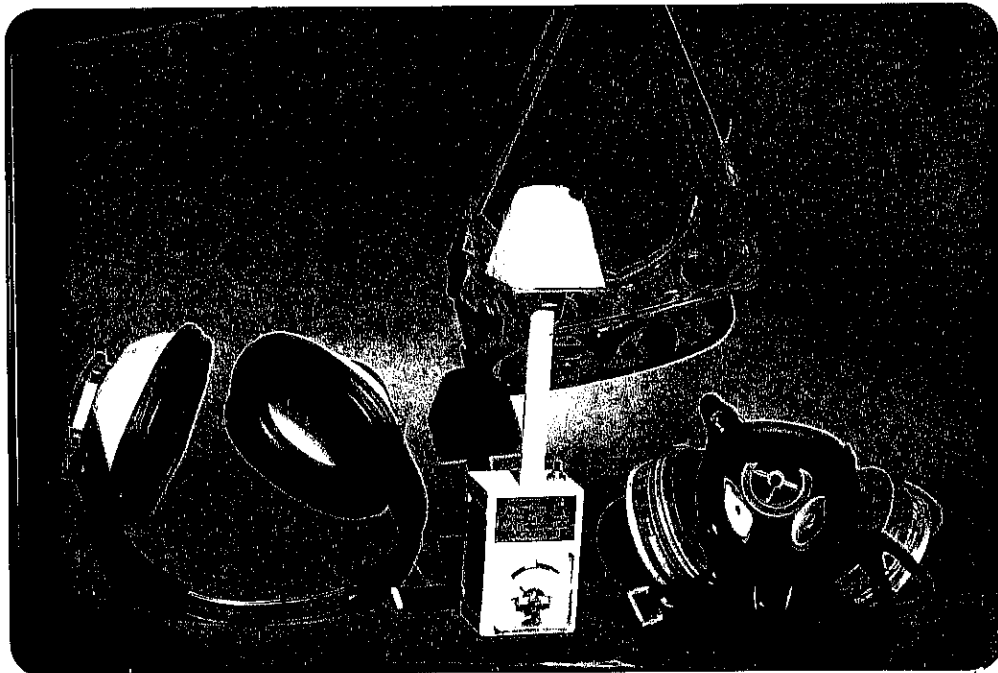


Figure 3. Safety equipment (1) meter to detect radiation leaks, (2) ear protectors, (3) goggles, and (4) air-purifying respirator

Two special safety steps are necessary before any work begins. They are (1) ascertain whether any crew member wears a pacemaker, and (2) whether any crew member has a heart or lung problem. The microwave can interfere with the pacemaker, and the solvent, Methylene Chloride, has been identified as a possible danger for people with a heart or lung problem.

#### Microwave Power Generator

The microwave power generator (MPG), sometimes referred to as the applicator, is a highly sophisticated piece of equipment. For a deeper understanding of the technical functions of the applicator the reader is referred to SRC Report SURC TR 76-071 (3).

As a prototype piece of equipment, problems with the applicator were not unexpected. However, most of the trouble with the applicator can be traced to two items in each unit. They are the circulators and the cooling system.

The circulators are ferric-magnetic discs designed to protect the magnetron from reflected radio frequency energy. They are so designed that any reflected RF energy in the wave guide would be directed into a dummy chamber where it could be dissipated without damage to the magnetron. They are so situated (Figure 4) in each unit that inspection of them is difficult. Only after many hours of maintenance and when a burned-out magnetron was being replaced were the circulators discovered to be faulty. The discs had become unglued, which allowed them to drop out of their intended position, allowing reflected RF energy back into the magnetron, and this is believed to be a major source of the problems. When it was discovered that the discs were coming unglued, the circulators were removed and returned to the manufacturer for rebuilding.

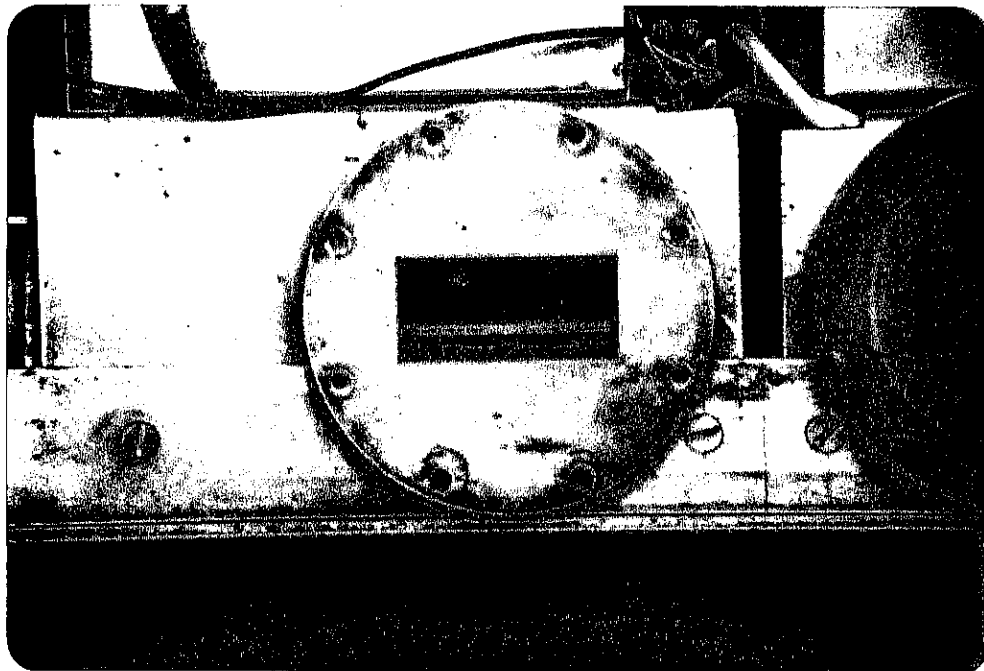


Figure 4. Cross section view of wave guide. Ferric ceramic circulator disks are inside rectangular-shaped wave guide

Five of the eight units in the applicator had defective circulators. The faulty circulators apparently caused most of the magnetrons to burn out. In all, seven of the original magnetrons burned out and were replaced.

The cooling system inside the applicator is made up of plastic and copper tubing. The tubing carries the coolant that circulates in the magnetron to keep it from overheating.

The cooling system occasionally would develop pinhole leaks and leaks at connections, which would cause electrical shorts in different components. Replacement of the plastic tubing reduced but did not eliminate all leaks. Interior views of the MPG are shown in Figure 5.

Some problems with the power supply were encountered. They occurred in the form of arcing or shorting out of the diodes. This may have been associated with leaks in the cooling system. However, extra insulation on the power supply mounting brackets reduced the frequency at which the diodes shorted out.

Even though some of the problems were major in scope they were correctable. Downtime for maintenance was extensive, primarily due to parts acquisition. Once it was determined that a major component such as a magnetron had failed, the time required to obtain that part could be five months. To alleviate that problem, after it was discovered that the magnetrons probably would fail one by one due to previous damage, replacement magnetrons were ordered in advance of need.

Other problems developed from time to time with the diesel generator and truck, but they were what could be considered normal and had no direct result in evaluation of the total system.



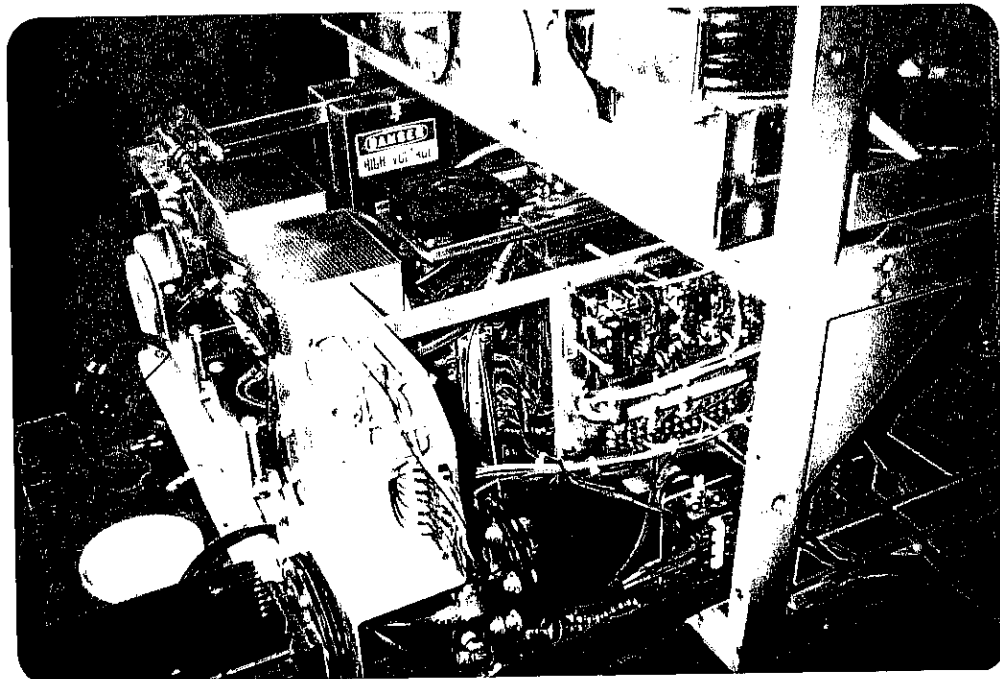
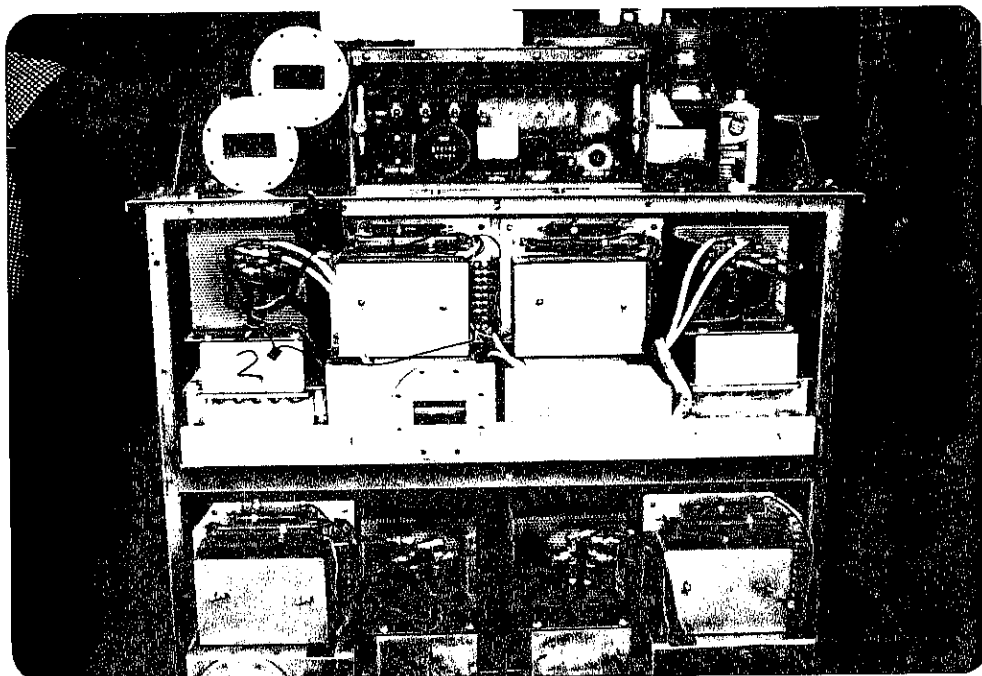


Figure 5. Two views of Microwave Power Generator (MPG) with side panels off during maintenance

## LABORATORY EXPERIENCE

As this was primarily a field study, laboratory work was kept to a minimum. Laboratory work was conducted only to acquaint the observer with some of the characteristics of the polymer concrete. As particular problems with the mix arose in the field, some work in the laboratory was done in order to find a solution to the problem. In one case the idea of in situ repair was tried in the laboratory to see if it could work in the field.

A problem encountered in the laboratory involved the use of proper equipment to handle the mix. The polymer concrete sticks to most laboratory equipment and must be washed in a solvent before it polymerizes. Paper or plastic throwaway containers were found to be practical. Curing of sample cores was done in 2-inch (50.8-mm) or 3-inch (76.2-mm) plastic pipe. Radiation time in the microwave oven was closely monitored so the cores could be withdrawn and extracted from the pipe before complete polymerization had developed. This procedure may have had some effect on the compressive strength of the cores made. However, they were high enough that damage of the cores during extraction was not considered.

When the manufacturer of the recommended catalyst was reluctant to sell in small quantities, an alternate source of a similar catalyst was found. According to the manufacturer the alternate catalyst would work equally well and they supplied samples of two types for testing. They were liquid, and when received one sample was frozen. After thawing, it would not produce a satisfactory product. The unfrozen sample was used and compared to the recommended catalyst.

Two samples were made, with the only variable being the type of catalyst. Figure 6 shows the average temperature curve of two cores after four minutes of radiation in the laboratory microwave oven and four minutes after removal of the cores before the first measurement was made. One core of each sample was tested under compression 40 minutes after removal from the oven. The core made with the recommended catalyst broke at 2930 psi (20.2 MPa) and the core made with the liquid catalyst broke at 3885 psi (22.0 MPa). Twenty-four hours later the second cores were broken with equal results, 3822 psi (26.4 MPa).

Even though the liquid catalyst was not used in the study, an alternate catalyst was available if needed.

The amount of resin per batch and the amounts of polyester and styrene were changed at times. The initial recommendation called for the resin system to be 60 percent polyester and 40 percent styrene by weight. To this was added 2 percent catalyst by weight. This resin system was then to be added at a rate of 10 percent by weight of the aggregate.

This mix seemed very rich, and some swelling of the patches was encountered. Laboratory tests revealed that by increasing the polyester and reducing the styrene, swelling could be reduced but not eliminated. Three blends were tried for comparison of strength and swell. The samples were made in 6-inch (12.7-mm) by 3-inch (6.4-mm) cylinders.

Polyester	<u>Percent</u>	Styrene	<u>Compressive Strength</u>	<u>Total Swelling</u>
			PSI	
60		40	6200	1/8-inch
70		30	6500	1/16-inch
80		20	7900	1/16-inch

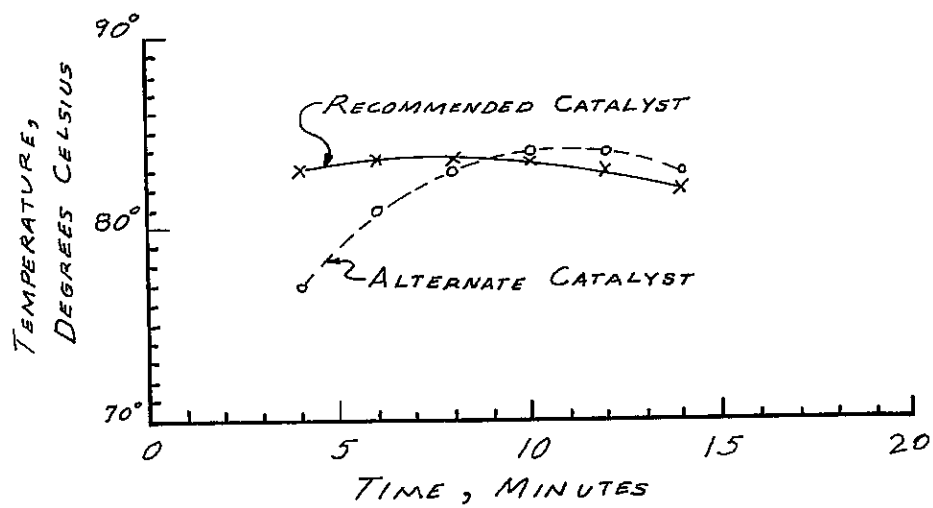


Figure 6. Surface temperatures of two samples with different catalyst. Elapsed time after removal from microwave oven

These tests were all with 2.0 percent catalyst and 8 percent resin by weight of the aggregate. After consultation with SRC, the resin system was changed to 70 percent polyester and 30 percent styrene.

While the 80-20 blend gave higher strength, the 70-30 blend was considered adequate for patching. Also, the 80-20 blend had a higher viscosity and required longer mixing. The 70-30 blend worked well in warm weather but became thicker in colder weather. Therefore it was decided to use the 60-40 blend when patching in cold weather. Even this blend became thicker at lower temperatures but was manageable.

The in situ-type patch was not specifically stated in the objectives but was seen as a possibility by such a versatile product. The in situ-type patch is defined as the repair of a badly broken piece of pavement or delaminated bridge deck without removing the broken concrete. In each case, the condition of the distressed area was considered critical, that is, a distressed pavement in situ repair should be made before pieces are dislodged and no pumping is present. Three in situ-type patches were made, with the best performing patch lasting 5 months under Interstate traffic.

To initially investigate the merit of the in situ repair concept, the idea was tested in the laboratory. Four PCC beams that had been tested previously in freeze-thaw cycles were selected. The dimensions of the beams were 16 inches (406.4 mm) long by 4 inches (101.6 mm) wide by 3 inches (76.2 mm) deep. Cardboard containers lined with wax paper were made to hold each beam. A mixture with the following proportions was made:

17.4% polyester

12.9% styrene

0.4% catalyst

50.8% cement

18.5% sand

Following is a brief description of the beams and how they were handled. They were all broken with mid-point loading.

Beam No. 1. This beam had been subjected to 293 freeze-thaw cycles, at which time its relative dynamic modulus was 93.7 percent. The high modulus indicates it was a sound beam so it was broken intentionally at the mid point. The two pieces were placed in the cardboard box 0.18 inches (4.6 mm) apart to simulate a large crack. The crack was filled with the resin mixture and cured. The modulus of rupture was 440 psi (3034 kPa), and the beam broke in a new location.

Beam No. 2. This beam, which had been subjected to 262 freeze-thaw cycles with a relative dynamic modulus of 67.3 percent, had several hairline cracks. After placing the beam in the container, 80 ml of the resin mixture was poured on top of the beam. The beam broke with a modulus of rupture of 650 psi (4482 kPa). After breaking, seams of the resin mix could be seen throughout the beam.

Beam No. 3. This beam had been tested to 293 freeze-thaw cycles, and had a relative dynamic modulus of 95.8 percent. Hairline cracks were present, and this beam was soaked in

water overnight, and allowed to drain and air dry on the surface before 110 ml of the resin was poured on top of the beam. The modulus of rupture was 291 psi (2000 kPa). The beam broke near the middle. The break was partially on an old crack and partially in adjacent material. The resin did not adhere on one side of the crack in the area where part of the break occurred.

Beam No. 4. This beam had been tested to 150 freeze-thaw cycles, and had a relative dynamic modulus of 53.4 percent. This beam was also soaked overnight in water, and allowed to surface dry before 220 ml of the resin mixture was poured on top of the beam. The modulus of rupture was 475 psi (3270 kPa), and the break occurred in a part of the beam where no visible crack was evident prior to applying the resin.

With these results it was felt that the in situ-type repair could be made with a reasonable degree of success, and it would be tried when possible.

Excessively moist (surface wet) aggregate may be used with a change of mixing procedure. When the first surface-wet aggregate was encountered, the mixing sequence was coarse aggregate, sand, cement, and resin. The mix looked good but the patch started raveling in a few days. Duplicating the conditions in the laboratory produced a sample that also raveled. It was determined that for wet aggregate this sequence of mixing coated the aggregate with cement and did not allow the resin the

necessary surface for bonding. To overcome this condition a change in sequence was tried.

The coarse aggregate and one half the resin were mixed together. Then the sand and one fourth the resin were added and mixed. The cement and remaining resin were added and mixed. This produced a good quality material in the laboratory that did not ravel. When it was tried in the field, the mixing times were 60 seconds, 30 seconds, and 15 seconds after each introduction of resin. The result was a quality mix that performed very well. Thereafter, this sequence was used whenever surface-wet aggregate was used.

#### FIELD EVALUATION

To evaluate the system the type of patches outlined in the objectives of the study were placed. An additional type of patch (in situ) was tried to further test and evaluate the system.

A total of 70 patches were made. Thirty-nine were made with crushed stone as the large aggregate and 28 were made with pea gravel as the large aggregate. Three in situ patches were made. Twelve patches failed. Twenty-two patches were removed from the study during extensive maintenance work or rehabilitation of the adjacent area. The three in situ patches failed. This section covers the field operation of the study.

#### Procedures

Patching with polymer concrete is very similar to patching with portland cement or asphalt cement. Traffic protection and preparation of the area to be patched are the same. Some crews prefer partial-depth sawing the limits of the area to be patched while other crews prefer only



the jackhammer to outline the area. Both types of area preparation were used, depending on the crew, availability of a saw, condition of the area to be patched, and its location.

After an area is cleaned out and ready for patching, the hole is painted with the resins before filling the hole with the polymer concrete. This allows some of the chemicals to penetrate the adjacent concrete, and makes a better bond between the patch and adjacent concrete.

At first a separate batch of resin and catalyst was mixed for painting the holes. Powdered aluminum was added to the resin at the rate of one pound powder to 40 pounds resin.

The idea of adding the powdered aluminum to the material used to paint a hole is that it will reflect the radiation back into the polymer mix, thus aiding temperature rise and reducing radiation time. This is correct, but without sensitive equipment the time required to cure a patch, with or without aluminum powder in the paint, could not be differentiated.

The first batch of paint, with the catalyst, polymerized within a few days after it was mixed. The next batch was mixed without the catalyst and remained fluid. The first batch probably polymerized due to either overexposure to direct sunlight or high ambient temperature. When it was determined that no measurable difference in curing time could be determined between patches painted with or without the aluminum powder, the paint as a separate item was eliminated. Holes then were painted with the same resin used for mixing with the aggregate.

The polymer concrete then was mixed in batches and placed in the hole. The batch size was about 1.5 cubic feet ( $0.04 \text{ m}^3$ ). Tamping the

mix was recommended and this was done for the first four patches. The mix stuck to the hand tamper and made finishing the patch difficult.

The next few patches were made by spading the mix in the hole to obtain consolidation, and finished with a hand float and trowel. This gave a better-looking patch and was used for the remainder of the study.

The polymer mix has a consistency very similar to cold mix and does not trowel easily. However, with some effort, enough matrix can be worked up to produce a tight surface. Obtaining a tight surface around the edge of the patch, with a trowel, is very difficult, but with some effort a tight surface over the entire area of the patch can be produced.

The polymer mix designed by SRC is adaptable to at least two different methods of handling, that is, it may be centrally mixed and transported to the work site or it may be mixed in the field. Central mixing was not used during the study for two reasons. First, since the unit was to be used in three districts covering a large part of the State, it was not practical. Second, and more important, it is more economical to mix in the field after the holes are prepared in order to mix only the quantity needed.

Initially, small quantities of the polyester, styrene, and catalyst were mixed in plastic buckets and hauled to the work site to be mixed with the aggregate. However, the chemicals not used would polymerize within a few days even when returned to a cool environment. This procedure was used because the supplier of the catalyst was apprehensive about mixing the catalyst out of doors where strong winds are possible. (Airborne dust from the catalyst can explode.) The catalyst dissolves

well in styrene but not in polyester. Therefore, the catalyst must be dissolved in the styrene before adding it to the polyester.

In order to eliminate waste of the chemicals by polymerization, the following procedure was used during most of the study. The polyester and styrene were mixed to the desired proportions except that one quart of styrene per batch quantity was withheld to be used to dissolve the catalyst. The polyester and styrene mix was transported in a 55-gallon (208.2-l) barrel. The styrene for dissolving the catalyst was kept in a 5-gallon (18.9-l) can. The catalyst was pre-measured indoors and placed in metal cans with airtight lids. The chemicals and catalyst were measured by using one of the charts in Appendix A. The cans of pre-measured catalyst were kept in a refrigerator until needed. To transport the catalyst in the field during warm weather the cans were placed in an insulated cardboard box along with a sealed container of ice. As each batch was mixed, one can of catalyst was dissolved in one quart of styrene, which was then added to the chemicals. If there was any wind, care was taken to open the catalyst in a sheltered place. The cab of a truck is a good place. This procedure worked well and no problems were encountered with handling the catalyst.

Mixing the polymer concrete was done in the same manner as portland cement concrete. The coarse aggregate, sand, and cement were added to the mixer and sufficiently mixed in 15 to 30 seconds. Then the chemicals were added slowly to prevent splashing. Mixing was continued for 30 to 60 seconds after all the chemicals were added. The convenience of the small portable mixer, seen in Figure 7, is that it can be placed next to the hole for dumping directly into the hole. Handling of the polymer

concrete should be kept to a minimum to eliminate unnecessary cleanup work after mixing.

A few gallons of the solvent Methylene Chloride were used to clean the mixer and hand tools. This solvent evaporates very fast and should be kept in an airtight container. After cleaning the mixer and tools, the solvent should be saved for reuse.

After the hole had been filled and troweled, the surface around the patch was swept clean. This is important to prevent radiation leaks. A small stone can keep the applicator from coming in contact with the pavement.

When placing the applicator over a patch, care should be taken to prevent the seals of the applicator from coming in direct contact with the mix. To prevent this situation, a supply of dry sand should be kept handy. With the applicator a few inches above the patch, sprinkle a thin layer of sand on the patch where the applicator will touch. This prevents the applicator from being glued to the patch as it cures. Sand also should be sprinkled on any area on the adjacent pavement that may have some residue of the mix that will come in contact with the applicator. A thin film of resin on the pavement without sand is sufficient to glue the flexible shields to the pavement during curing. Figure 8 is a view of the underside of the applicator showing the slotted wave guides and flexible shielding around the perimeter.

When the applicator is in position the remote control switch is plugged in and the operator turns on the unit. A check is then made for radiation leaks. If any are found, sandbags are placed at that location and rechecked. Occasionally leaks did occur and were reduced

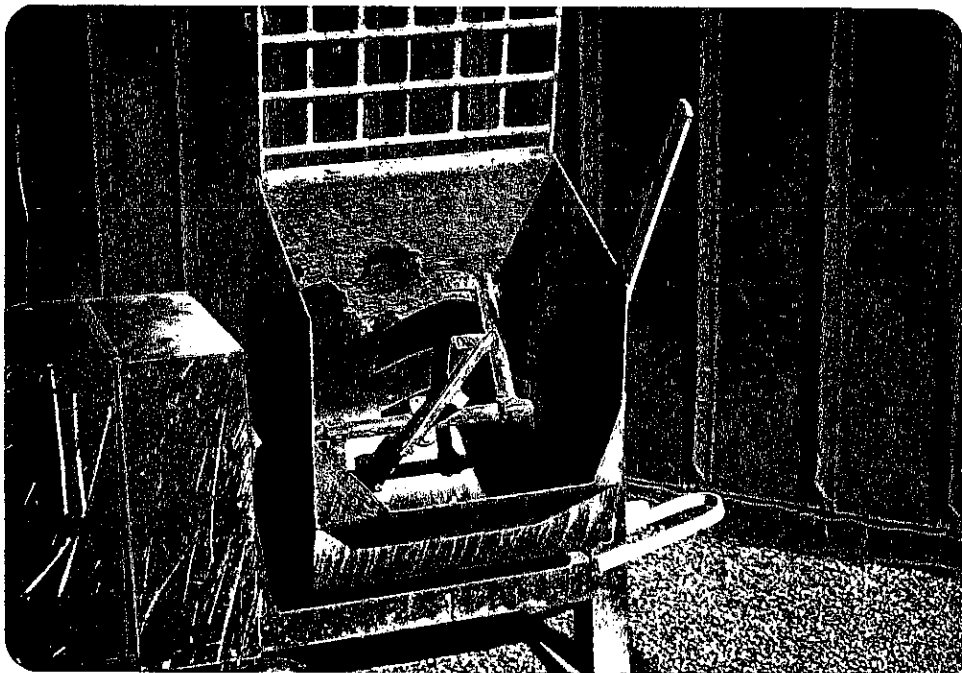


Figure 7. Small mixer used to mix polymer concrete

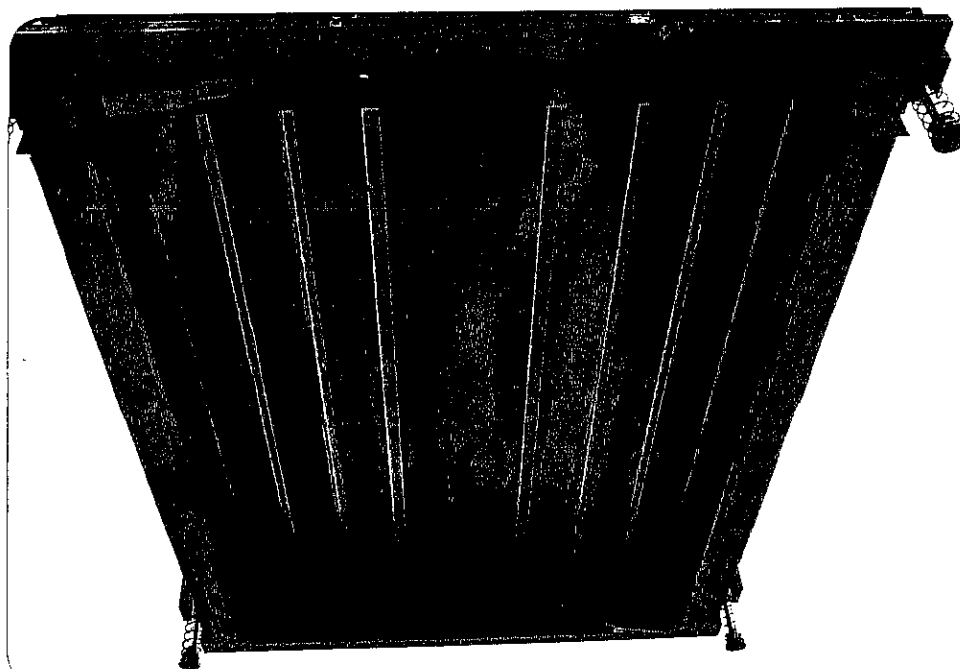


Figure 8. Bottom view of applicator showing slotted wave guides which distribute microwaves over patch, and flexible shielding

to a safe level or eliminated with sandbags. The applicator (MPG) is seen being unloaded and set in position over a patch in Figures 9 and 10.

The total time from beginning to completion of a patch can vary greatly for several reasons. To evaluate this system, only radiation time was recorded. Because traffic protection and preparation of the area are common to most types of patching, these were not considered. With all parts of the system at an optimum condition the time required to cure a patch depends on its size. A patch no larger than the applicator could be cured in as little as 10 minutes. Larger patches, requiring additional setups, would take proportionately longer. When the polymerization temperature is reached the polymerization process is very rapid. Most patches were cured and ready for traffic when the applicator was removed. Ideally, the applicator should be removed when the exothermic reaction of polymerization starts. Because this point was not identifiable in the field, the tendency was to over-radiate the patch.

#### Patches

Bridge deck patches far outnumber other types of patches made. One reason for this was the crews used were the bridge maintenance crews and they were more familiar with the problems on bridge decks. Another reason is that the patches on bridge decks generally were smaller and several patches could be made each day.

Table 2 lists all patches. They are identified by patch number, type of patch, type of coarse aggregate, percent of resin by weight per batch, date placed, and date failed or removed.



Figure 9. Microwave Power Generator being unloaded from truck



TABLE 2. LIST OF PATCHES MADE DURING FIELD EVALUATION

Patch No.	Type of Patch <sup>1/</sup>	Type of Coarse Aggregate		Percent of Resin	Date Placed	Date Failed or Removed
		Cr. St.	Pea			
1	2	x		10.0	08-12-77	-
2	2	x		7.4	08-12-77	-
3	2	x		7.4	08-12-77	-
4	2	x		7.4	08-12-77	-
5	2	x		7.9	08-15-77	Oct. 1978*
6	2	x		7.9	08-15-77	-
7	2	x		7.9	08-15-77	-
8	2	x		7.9	08-15-77	Oct. 1978*
9	2	x		7.9	08-15-77	Oct. 1978*
10	2	x		7.9	08-15-77	Oct. 1978*
11	2	x		7.9	08-15-77	Oct. 1978*
12	2	x		7.9	08-15-77	Oct. 1978*
13	2	x		7.9	08-15-77	Oct. 1978*
14	2	x		7.9	08-15-77	Oct. 1978*
15	2	x		7.9	08-15-77	Oct. 1978*
16	2	x		7.2	08-30-77	Oct. 1978*
17	2	x		7.2	08-30-77	Oct. 1978*
18	2	x		7.2	08-30-77	Sept. 1977
19	2	x		7.2	08-30-77	-
20	2	x		7.2	08-30-77	Dec. 1977
21	2	x		7.2	08-30-77	Oct. 1978*
22	2	x		7.2	08-30-77	Oct. 1978
23	2	x		7.2	08-30-77	Oct. 1978*
24	2	x		7.2	08-30-77	Oct. 1978*
25	3	x		7.2	09-19-77	Sept. 1978
26	1		x	7.0	09-26-77	-
27	1		x	7.0	09-28-77	-
28	1		x	7.0	10-03-77	July 1980*
29	2		x	7.4	10-04-77	-
30	1		x	7.0	10-05-77	-
31	5		x	7.0	10-11-77	Oct. 1979
32	5		x	7.0	10-12-77	-
33	2		x	7.1	11-16-77	-
34	2	x		8.0 & 8.6	07-07-78	Aug. 1980*
35	2	x		9.8	07-07-78	Aug. 1980*
36	2	x		9.8	07-07-78	Aug. 1980*
37	2	x		9.8	07-07-78	Aug. 1980*
38	3		x	7.0	09-19-78	Dec. 1978
39	3		x	7.0	09-20-78	Dec. 1978
40	6	-	-	-	09-20-78	May 1980
41	2		x	11.4	09-26-78	-
42	2		x	11.4	09-26-78	-
43	2		x	11.4	09-26-78	-
44	2		x	11.4	09-26-78	-
45	2		x	11.4	09-26-78	-

TABLE 2. LIST OF PATCHES MADE DURING FIELD EVALUATION (Cont'd.)

Patch No.	Type of Patch <sup>1/</sup>	Type of Coarse Aggregate		Percent of Resin	Date Placed	Date Failed or Removed
		Cr. St.	Pea			
46	2		x	11.4	09-26-78	-
47	2		x	9.8	09-26-78	-
48	2		x	11.4	09-27-78	-
49	2		x	11.4	09-27-78	-
50	2		x	11.4	09-27-78	-
51	2		x	11.4	09-27-78	-
52	2		x	11.4	10-19-78	-
53	2		x	11.4	10-19-78	-
54	2		x	11.4	10-20-78	-
55	2		x	11.4	10-20-78	-
56	2		x	11.4	10-20-78	-
57	2		x	11.4	10-20-78	-
58	6	-	-	-	10-24-79	Jan. 1980
59	2		x	11.0	11-06-79	Oct. 1980*
60	6	-	-	-	01-29-80	May 1980
61	4	x		9.8	06-26-80	Nov. 1980
62	3	x		9.8	06-26-80	Aug. 1980
63	3	x		9.8	06-26-80	-
64	3	x		9.8	06-26-80	Oct. 1980
65	4	x		9.8	06-26-80	Nov. 1980
66	4	x		9.8	06-27-80	-
67	4	x		9.8	06-27-80	Aug. 1980
68	4	x		9.8	06-27-80	Sept. 1980
69	4	x		9.8	06-27-80	-
70	4	x		9.8	07-01-80	Sept. 1980

<sup>1/</sup> Type of Patch

- 1 - Full-depth bridge deck
- 2 - Partial-depth bridge deck
- 3 - Full-depth CRC pavement
- 4 - Partial-depth CRC pavement
- 5 - Full-depth contraction joint
- 6 - In situ repair

\* Patch removed but not failed

Patches 1 through 15, 59, and 60 were made with 60 percent polyester and 40 percent styrene. All other patches were made with 70 percent polyester and 30 percent styrene. The patches made in 1977 had 2 percent catalyst and all others had 1.5 percent catalyst.

### Discussion

Quality control of a mix starts in the laboratory but is adjusted in the field. The recommended surface-dry aggregate gives the optimum conditions for troweling and curing of the mix. This condition of the aggregate seldom is found in the field. When truly surface-dry aggregate is available, the amount of internal moisture is hard to determine in the field. The internal moisture is required to create the temperature rise necessary to initiate the polymerization. Usually the aggregate has enough internal moisture to do the job. When the moisture is not present it is apparent because of the excessive time required to raise the temperature of the mix. Normally a temperature rise is noted after 4 to 5 minutes of radiation when sufficient moisture is present. Unfortunately, the absence of moisture in the aggregate is not apparent until radiation of the patch has started.

On one occasion when aggregate did not have sufficient moisture, the time required to raise the temperature of the patch was 45 minutes. For the next patch one pint of water was added per batch, and the time required to raise the temperature was reduced to 37 minutes. When two pints of water per batch were added to the aggregate, the time required to raise the temperature was reduced to about 25 minutes. The addition of such a small amount of water had no apparent effect on the quality

of the polymer concrete. It worked well and the patches still are in very good condition.

Determining when the applicator should be removed was a trial-and-error approach. At first the normal procedure was to radiate the patch a few minutes (5 to 10), lift the applicator, check the batch temperature, and radiate again if required. Usually the patch would be hard after the second or third radiation period. To eliminate the interim inspection of the patches, thermocouples were used on a few patches to monitor the internal rise in temperature. It was thought that by doing this the applicator could be left in place and lifted when the patch was hot enough to complete polymerization. Unfortunately, this was never accomplished. However, experience taught that by feeling the exhaust air from the cavity in the applicator over the patch and noting a change in odor of the exhaust air, the applicator could be left in position until the patch was hot enough to complete polymerization.

Factors that determine the length of radiation time other than moisture of the aggregate are ambient air temperature, temperature of adjacent pavement, wind speed, and mass of patch.

In the summer when these temperatures are high, the applicator can be removed before polymerization is complete, with good results. However, during low temperatures and high wind speed, removal of the applicator without protecting the patch can result in incomplete polymerization, which will require more radiating.

Even though internal patch temperatures could not be measured, the surface temperature was measured after the applicator was removed. Figure 11 shows some typical temperature curves. In all cases, the

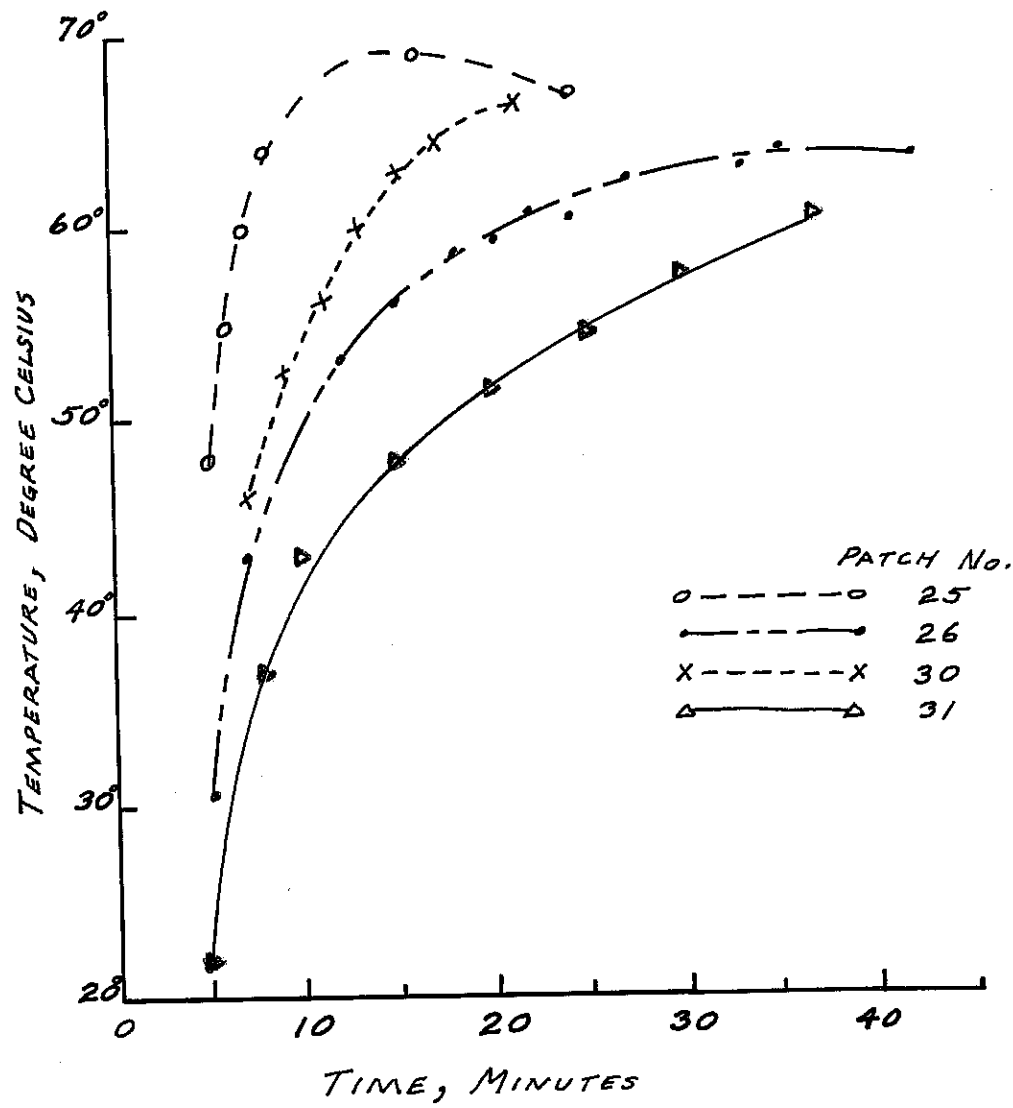


Figure 11. Typical surface temperature curves of four patches after radiation

low point on each curve represents the first reading after removal of the applicator; the time axis represents elapsed time after removal of the applicator. Additional information for the patches is given in the table below.

Patch No.	Type Agg.	Depth of Patch Inches	Water Added Pints	Radiation Time		Air Temp. °C
				Low Minutes	High	
25	Cr. St.	9	0	10	10	24
30	Pea	7	3	20	15	18
26	Pea	5½	0	20	25	24
31	Pea	11	0	20	30	9

These data point out the effect of some of the variables. The water was inadvertently left out of patch numbers 26 and 31. The patches made with crushed stone usually polymerized with less radiation. This probably is due to the crushed stone having more internal moisture. No water was added to the aggregate when using crushed stone. The shape, surface, and mineral content of the two coarse aggregates may account for some difference in radiation time. These curves also point out the exothermic nature of the polymerization reaction.

It is not always necessary to radiate the entire surface of the patch. During the summer the polymerization process will continue a few inches beyond the area radiated. However, at low temperatures this does not happen when the heat loss is so great that polymerization process cannot be sustained.

During patching in cold weather some insulating material such as burlap, kraft paper, or canvas should be kept handy to protect the patch from heat loss on the surface.

The amount of resin used per batch varied from time to time. Even though the developer had recommended 10 percent resin, this could be adjusted for different reasons. A good rule of thumb was that a good-looking mix would produce a good product. This rule often was followed, with good results.

The moisture in the aggregate had an effect on the mix other than the rise in temperature. The drier the aggregate, the more resin absorbed, especially with the crushed stone. The pea gravel didn't seem to absorb much resin even when it was dry. The crushed stone, however, could absorb enough resin to affect the mix. The resin absorbed by the aggregate may not affect the strength, although it is suspect, but it does affect its workability. When drier-than-normal crushed stone is suspected, the mix should be left in the mixer longer (up to 2 minutes). By doing this it could be determined whether the mix was going to be too dry from absorption and more resin should be added.

The different amounts of resin used did not seem to relate directly to the performance of a patch. Raveling may be more closely related to the amount of sand than the amount of resin. The amount of resin used for some patches was too low. An excess of resin is better than a deficiency for both workability and appearance when completed.

Swelling as determined in the laboratory was affected by the ratio of polyester and styrene. It also was found to be affected by the rate of heating the patch.

Steam often was seen coming out of small holes in a patch, especially under the slotted wave guide nearest the magnetron. This was believed to be due to radiating in only the high-power mode, which was the practice

at first. The intense RF energy probably caused the moisture to vaporize faster than the resins could polymerize and thus caused swelling in parts of a patch. To correct this, the practice of radiating the patch in the low-power mode for 8 to 15 minutes before switching to the high-power mode was followed. This gave the desired results of lowering the rate of temperature rise and allowing the patch to heat more uniformly, which eliminated the uneven swelling. An additional advantage of this practice was that any moisture in the applicator could be dried out in a mode that was not as likely to cause electrical shorts, which were a problem at times.

The only adjustments to the proportions of aggregate were done with the crushed stone aggregate mix. After examining the first 24 patches, which had 61 pounds (27.7 kg) sand per batch, it was apparent that more sand was needed in the mix. The sand was adjusted to 72 pounds (32.6 kg), which eliminated raveling. Figures 12 and 13 show two partial-depth bridge deck patches made with insufficient sand. These are the only patches declared failed when using the 61 pounds (27.7 kg) sand per batch. As a contrast, patch number 25, Figure 14, was made using 72 pounds (32.6 kg) sand per batch and had only very light raveling, even though it carried several times the traffic that patch number 20 carried. Patch number 25 was declared failed after one year of service. However, it was believed that the broken steel in the patch and the adjacent pavement failure contributed to failure of the patch.

Figure 15 shows a full-depth CRC pavement patch with 9.8 percent resin. The difference in appearance in surface texture between patch number 25 and patch number 63 is apparent. A closeup view of patch





Figure 12. Patch number 18 showing structural failure



Figure 13. Patch number 20 showing severe raveling

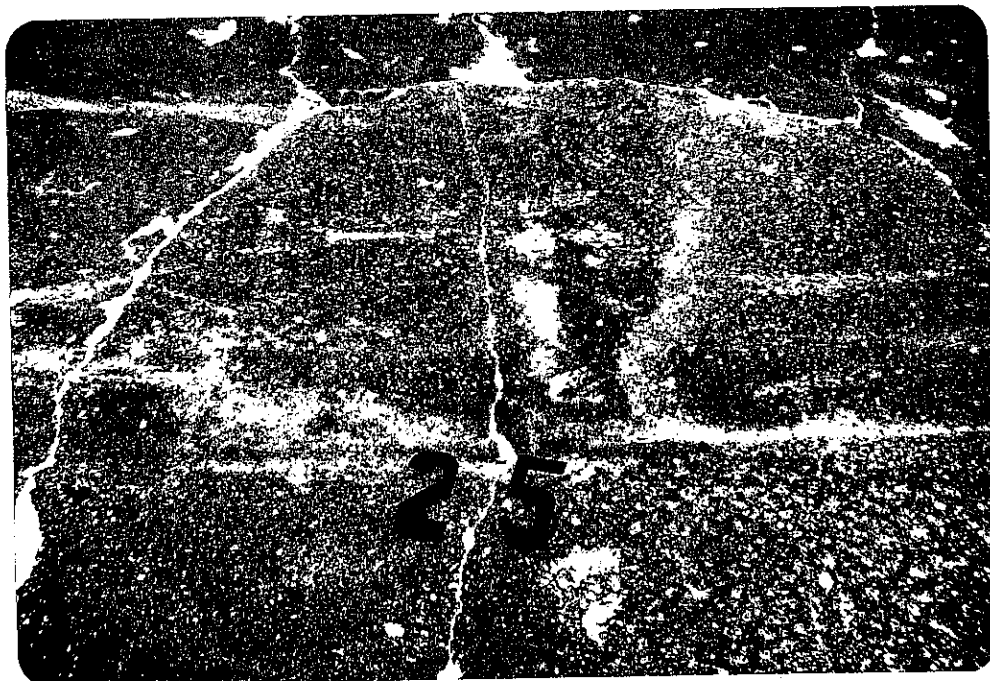
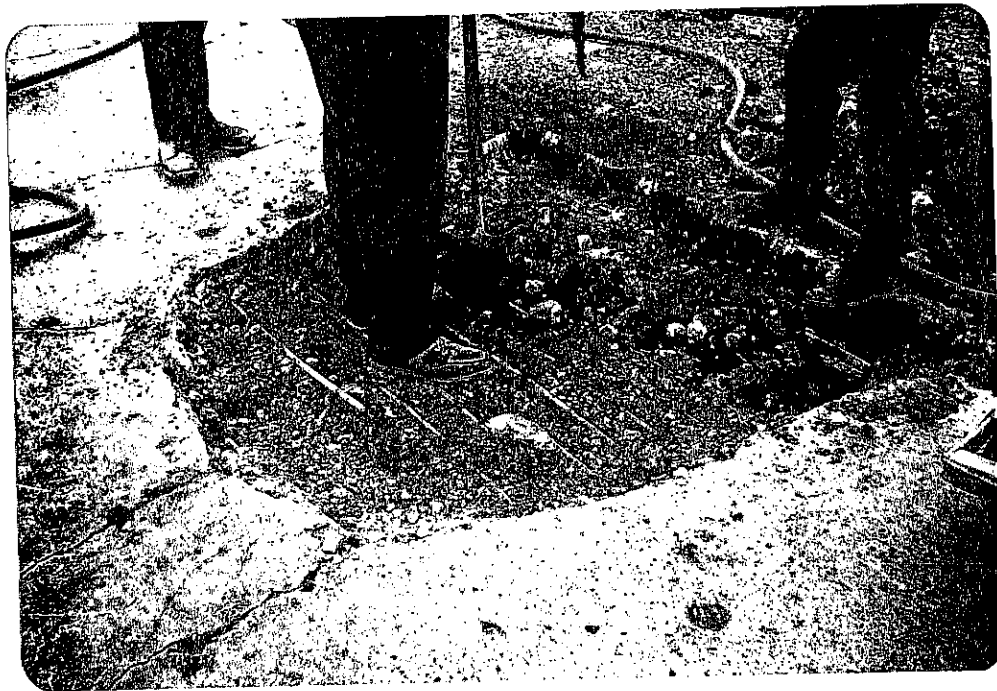


Figure 14. Full-depth CRC patch number 25 before and after one year service. Transverse crack is over broken steel seen in top photo

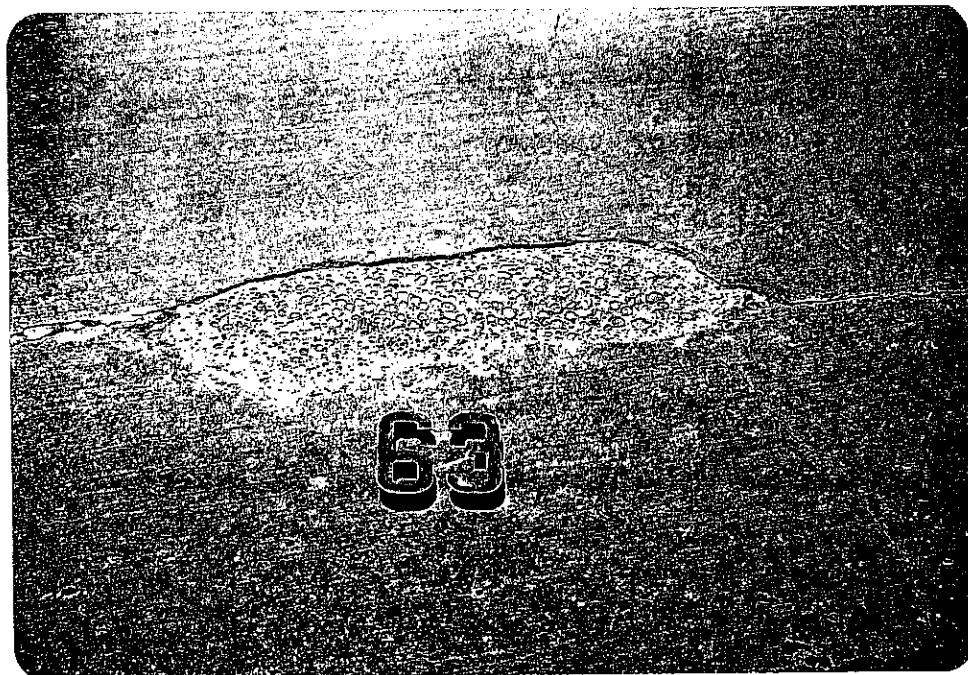


Figure 15. Full-depth CRC punch-out patch number 63 before and after

number 62 is seen in Figure 16. The moonlike craters do not make a good-looking patch, but sharp edges should provide good friction.

An example of a patch made with pea gravel is seen in Figures 17 and 18. The surface of a patch made with pea gravel appears more like regular portland cement concrete. This is a full-depth bridge patch with an excellent performance. The parallel depressions seen in the patch in Figure 17 are caused by the flexible radiation shields in the bottom of the applicator.

A full-depth contraction joint patch is seen in Figure 19. This patch is 3 years old and performing very well. Some spalling has developed on the edges. The raveled area in the outside wheelpath is the result of fire. The MPG broke down before the entire patch could be radiated. The outside four feet was heated with a 600,000 BTU "portable infrared asphalt heater." The heater was placed about 3 inches (76.2 mm) above the patch and it caught on fire. The fire was extinguished with sand and the heater replaced 6 inches above the patch without catching on fire again. Other than the raveling where the patch was on fire, there was no problem with this patch.

An example of an in situ-type repair is seen in Figure 20. While this type of repair did not yield the desired results, some things were learned by doing them. Patch number 40 in CRC pavement performed the best and it is believed it would have performed better except for the deterioration of the adjacent concrete. Patch number 58 was at the intersection of a transverse joint and the centerline joint. This distressed area had been pumping, and consequently the pieces were covered with a film that would not allow the resin to adhere. Patch number 60,

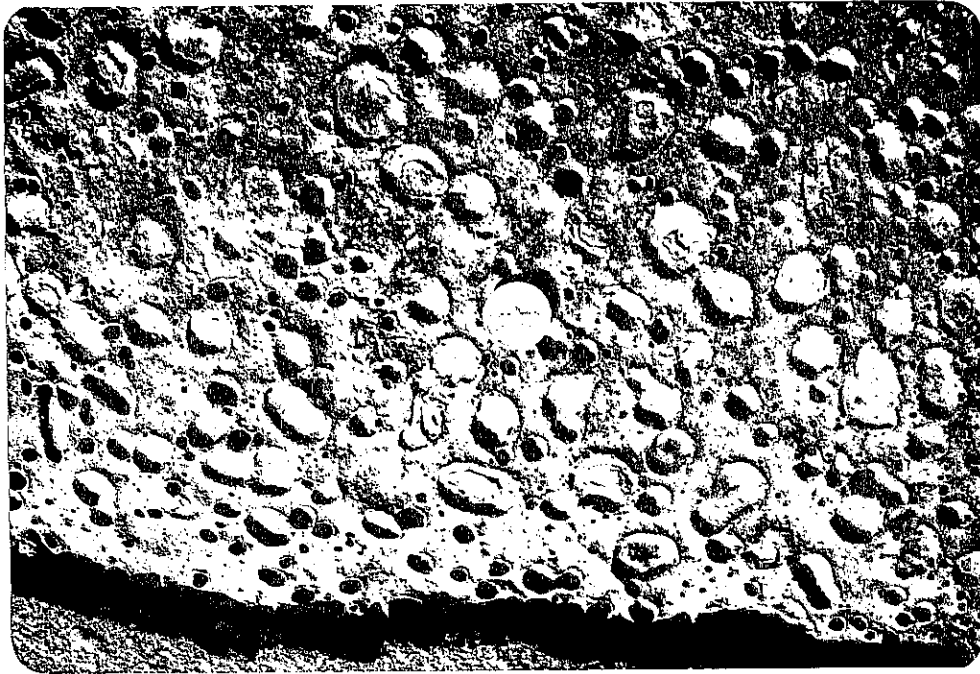


Figure 16. Close-up view of patch number 63 showing surface texture with moonlike craters



Figure 17. Patch number 27, three years old

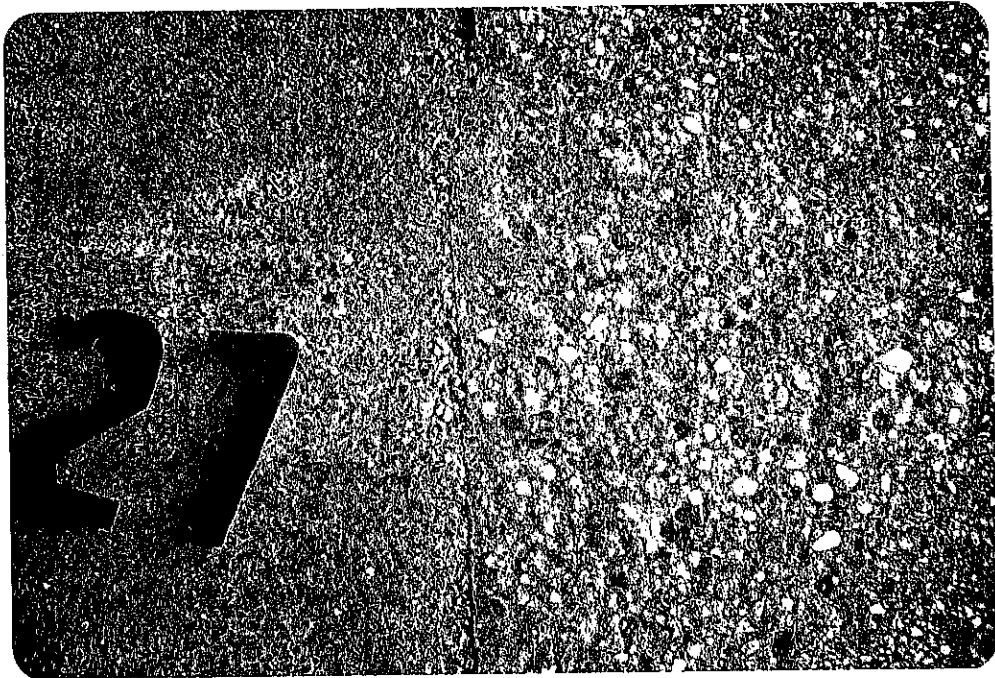


Figure 18. Close-up view of patch number 27  
on left showing tight surface



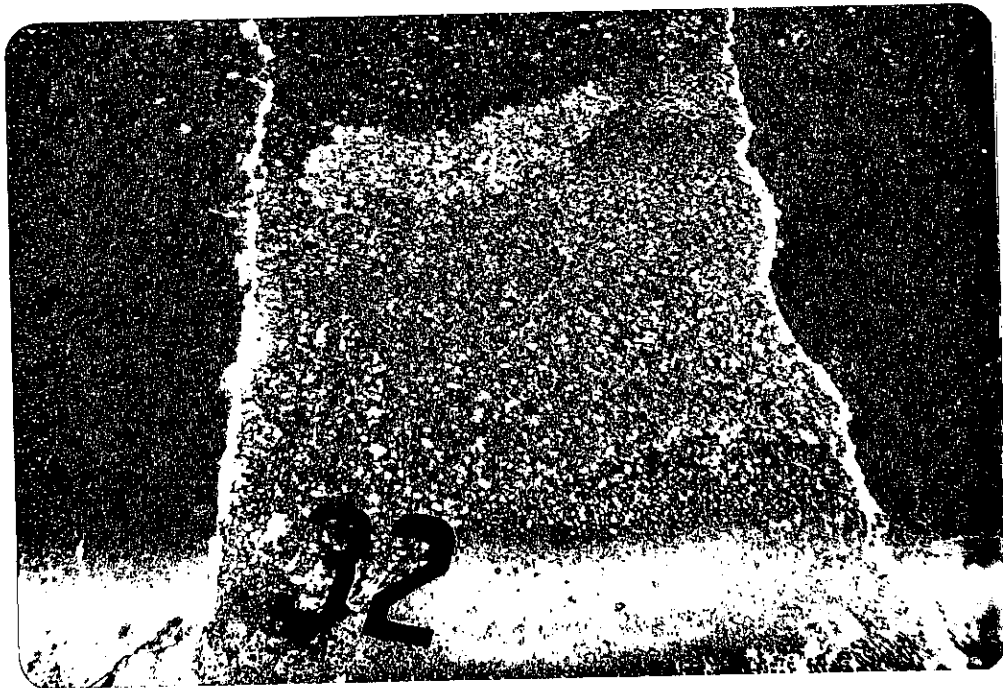
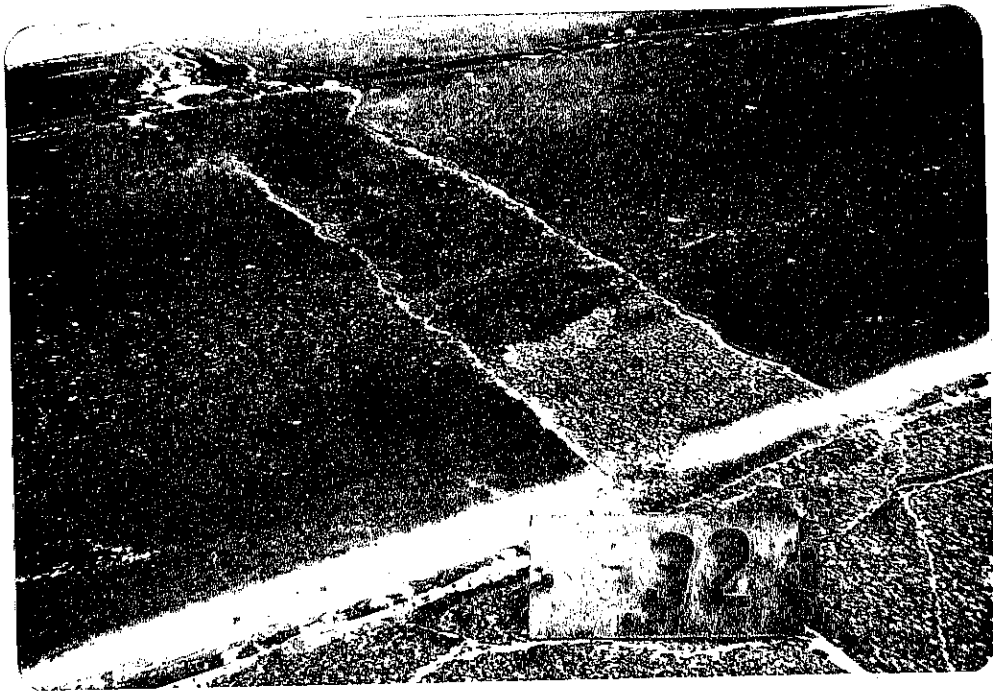


Figure 19. Full-depth contraction joint, patch number 32

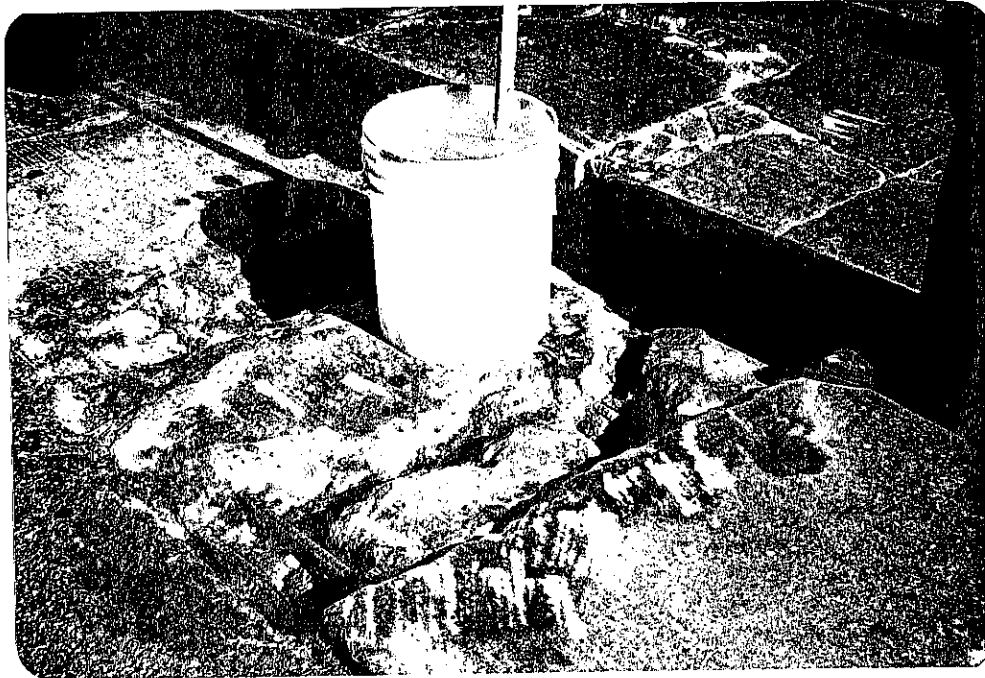


Figure 20. In situ patch number 60 before applying resin and morning after curing. White area is snow and ice on patch

also in CRC pavement, had been patched with cold mix, and not all the cold mix could be removed from the cracks. In all cases, the only means of getting the resin into the distressed area was to pour it over the area and allow it to penetrate the cracks as best it could. It is believed this type of repair could be successful if a positive means of injecting the resin could be developed.

Patch number 60 also was made during the coldest temperatures that the equipment was used. The air temperature was 16<sup>0</sup>F (-9<sup>0</sup>C) with a 10 M.P.H. wind. About 10 minutes more radiation per setup was required to bring the patch up to temperature to attain polymerization. In this case the exothermic nature of the reaction was not great enough to overcome the heat loss to the adjacent pavement and air, and polymerization was attained by leaving the applicator in position until polymerization was complete.

#### Physical Tests

Samples of several patches were taken for testing the air content and compressive strength of the material. As can be seen in Table 3, the range of air content is from 3.9 percent to 11.2 percent and the range of compressive strength is from 2732 psi (18,837 kPa) to 5137 psi (35,419 kPa). With these large differences there are no trends established when comparing air voids, compressive strength, or percent of resin in the samples.

The fact that 10 of the 12 patches that actually failed were made with crushed stone is not viewed as a result of the stone. Patch numbers 61 through 70 were patches of punch-outs on CRC pavement 2 to 5 years old. It is believed that the forces that caused the original failures probably would cause any material to fail until these forces are identified and neutralized.

TABLE 3. AIR CONTENT AND COMPRESSIVE STRENGTH OF SELECTED PATCHES

<u>Patch No.</u>	<u>Air Content Percent</u>	<u>Compressive Strength</u> PSI	<u>Percent of Resin</u>	<u>Date Failed or Removed</u>
7	-	5119	7.9	
21	4.0	-	7.2	Oct. 1978*
25	3.9	4554	7.2	Sept. 1978
26	-	5137	7.0	
27	8.0	4340	7.0	
29	-	4913	7.4	
30	8.9	3181	7.0	
32 IWP <sup>1/</sup>	6.7	3128	7.0	
32 OWP <sup>2/</sup>	7.2	2732	7.0	
36	3.9	4370	9.8	Aug. 1980*
37	6.3	4612	9.8	Aug. 1980*
59	11.2	4480	11.0	Oct. 1980*
61	9.5	4141	9.8	Nov. 1980
62	6.3	-	9.8	Aug. 1980
65	8.2	3882	9.8	Nov. 1980
70	4.7	3945	9.8	Sept. 1980

<sup>1/</sup> Inner wheelpath  
<sup>2/</sup> Outer wheelpath

\* Patch removed but not failed

During compressive tests it was noted that the crushed stone aggregate sheared and the pea gravel did not, indicating a better bond of the matrix to the crushed stone. The average compressive strength of the crushed stone samples was 328 psi (2261 kPa) higher than the average compressive strength of the pea gravel samples. Air voids did not seem to affect the strength of the samples.

#### Comparative Test

In order to further evaluate the system, comparative tests were made using regular portland cement concrete and proprietary mixes. The tests were made in all three districts. One was a joint repair in District 4 and partial-depth bridge deck repairs in Districts 3 and 6. The comparative patches are identified below with the type of material used, date patched, and date failed or removed from service.

<u>Patch Number</u>	<u>Material</u> <sup>1/</sup>	<u>Date Patched</u>	<u>Date Failed or Removed</u>
31A	PM-1	10-11-77	Oct. 1979*
51A	PM-1	09-27-78	-
51B	PM-1	09-27-78	-
51C	PCC	09-27-78	-
55A	PCC	10-20-78	Nov. 1978
57A	PCC	10-20-78	Nov. 1978
57B	PCC	10-20-78	Nov. 1978
59A	PM-2	11-06-79	Oct. 1980*
59B	PM-1	11-06-79	Oct. 1980*
59C	PM-3	11-06-79	Oct. 1980*
59D	PM-1	11-06-79	Nov. 1979

<sup>1/</sup> PM-1, PM-2, PM-3 designate different proprietary mixes  
PCC - portland cement concrete

\* Patch removed but not failed

Patch number 31A was placed when the air temperature was 48°F (9°C) and falling. When the patch was removed due to rehabilitation of the pavement it was showing signs of failure and in worse condition than the polymer concrete patch.

Patch numbers 51A, 51B, and 51C were placed when the air temperature was in the low 80's. These patches performed well and were covered with an asphalt material during the summer of 1979.

Patch numbers 55A, 57A, and 57B were placed when the air temperature was approximately 58°F (14°C) and falling. These patches probably were not cured properly before opening them to traffic because they failed within 15 days. Failure was in the form of severe scaling.

Patch numbers 59A, 59B, 59C, and 59D were made with three different types of proprietary mixes when the air temperature was 39°F (4°C). Patch number 59D failed within a few hours. The other patches performed well and were removed in October 1980 when extensive deck repairs were made.

A cost comparison showed that the polymer concrete is competitive when compared to proprietary mixes. Using costs in force when patch numbers 31 and 31A were made shows that the polymer concrete per cubic foot cured in place was 70 percent of the cost of the cheapest proprietary mix per cubic foot in place. This comparison would come closer to unity if aggregates were added to the proprietary mix and the polymer patch had a larger surface area, which would require additional setups for curing.

Due to the size of the applicator, large patches (greater than 8 square feet) are less efficient to make than small patches. However, the system as it exists has a high potential for producing good-quality patches in warm or cold weather. Because of the polymerization process, full strength is achieved in a short period of time after radiating the patch, usually by the time the equipment can be loaded by the crew.

This gives the system a high potential for patching in cold weather on high-volume highways where overnight lane closures are to be avoided.

While the data from comparative patches are not conclusive, they do point out the advantage of polymer concrete cured with microwave energy over PCC patches when the air temperature is in the range observed (39° to 58°F). Unfortunately, durability comparison over a longer period could not be made. The comparison patches, that survived the first few days, and polymer patches at a given location both were performing equally well when they were removed for more extensive maintenance.

#### CONCLUSIONS AND RECOMMENDATIONS

- (1) The resin system selected provides adequate strength and durability for patching highways.
- (2) Saturated surface-dry aggregate works best for the polymer concrete cured by microwave. Water may be added to very dry aggregate to reduce the radiation time.
- (3) Surface-wet aggregate requires a change in the order of mixing to produce an adequate material.
- (4) A short training session should be held for the crew before any work begins, and all safety procedures followed.
- (5) The tubing in the applicator should be relocated to minimize the effects when leaks do occur.
- (6) The ratio of polyester and styrene should be adjusted for cold-weather patching.
- (7) Swelling, while not a serious problem, may be kept to a minimum by adjusting the resin ratio and by bringing the patch temperature up slowly.

- (8) Protecting the patch from heat loss is necessary in cold-weather patching.
- (9) The system can produce quality patches competitive with existing proprietary mixes.
- (10) The system has the greatest potential for cold-weather patching on high-volume highways when overnight lane closures are to be avoided.
- (11) The system should be turned over to one of the crews with experience to be used for patching.



## REFERENCES

- (1) Sawyer, Richard G., Boyko, Leo L., and Hunt, George M., Microwave Heating for Road Maintenance, Syracuse University Research Corporation, Final Report SURC TR 73-436, August 1973.
- (2) Boyko, Leo L., Lederer, E. H., and Sawyer, Richard G., Microwave Heating for Road Maintenance, Syracuse University Research Corporation, Final Report SURC TR-76-052, March 1976.
- (3) Boyko, L. L., Lederer, E. H., and Sawyer, R. G., Microwave Road Patching System Model Exp.-2 Operations and Maintenance Manual, Syracuse University Research Corporation, Report SURC TR 76-071, October 1976.
- (4) Rapid-Setting Materials for Patching of Concrete, NCHRP Synthesis of Highway Practice 45 (1977), 13 pp.
- (5) Kukacka, L. E., Fontana, J., and others, Concrete Polymer Materials For Highway Applications, Brookhaven National Laboratories, Progress Report No. 3, May 1974, 141 pp.

## APPENDIX A

This appendix includes a sequence of operation for the generator, cooling system, and microwave unit. The equipment operator should be provided with this sequence for ready reference in the field. The shutdown sequence (No. 17, 18, and 19) is important to follow so that the startup sequence may be properly followed.

The charts (Figures A-1 and A-2) are a convenient means to aid mixing the chemicals to get the correct quantities. These are for the two proportions used during the study. The metering works well when translucent plastic buckets are used with marks on the outside in tenths of a gallon. The charts may be used for weight or volume. The catalyst should be weighed in grams if possible. A small laboratory scale works well.

Material safety data sheets for each chemical used are included here and should be provided by the manufacturer when the chemicals are purchased. A copy should be kept with the equipment for reference if needed.

- (1) Start Primary Power Generator (PPG). Instructions are displayed on the PPG Control Panel Cover.
- (2) Connect power plug from PPG to receptacle located on the top of Microwave Power Generator (MPG).
- (3) Connect coolant hoses (2) from cooling system to MPG quick disconnect hose couplings (IN to IN).
- (4) Place MPG on the area to be illuminated.
- (5) Switch Primary Power ON (Lever Switch on PPG), and adjust Primary Voltage to 208V (Red Mark on the Voltmeter).
- (6) Switch cooling system ON (Cooling fan and pump).
- (7) Set Circuit Breakers (CB) on individual Microwave Power Units (MPG) to ON position.

- (8) Set Power Unit's ON-OFF switch (MPG - Control Unit) to ON position.
- (9) Set Hi-Lo switch (MPG - Control Unit) to LO position.
- (10) After "Ready Light" is actuated (approximately 10 sec.), observe Field Coil current on Magnetron Current Meter (MPG - Control Unit). Current should be approximately 640 ma (6.4 V on the meter). Check all eight units utilizing magnetron current selector switch (MPG - Control Unit).
- (11) Set System Control Unit Power ON-OFF Switch to ON position (MPG - Control Unit).
- (12) Check INTERLOCK on indicating light (should be ON).
- (13) Keep safe distance from MPG (approximately 10 feet), and set REMOTE RADIATE Switch (hand-held) to ON position. RADIATE Indicator Light on top of MPG should be ON (Flashing Light).
- (14) Measure leakage level with Leakage Monitor. Slowly introduce the thermocouple probe into the unknown field. Hold the probe by its handle and point toward the unit under test. The meter should be held away from the suspected radiating source. Move the probe in a circular motion around the suspected area of radiation leakage. Observe the meter reading.

### C A U T I O N

OBSERVE THAT METER READING DOES NOT  
EXCEED FULL-SCALE DEFLECTION

If Leakage Monitor indicates excessive radiation level (more than  $1\text{mW}/\text{cm}^2$  - 5 cm) from the periphery of the applicator, set RADIATE switch to OFF position, and examine the adherence of the applicator to the roadbed. Any gaps between the applicator's enclosure and the roadbed should be covered with sand or microwave-absorbing material.

- (15) As per procedure 14, if RF leakage is less than  $1\text{mW}/\text{cm}^2$ , check Magnetron Current (magnetron current meter and associated switch) on all eight power units. Current should not exceed 560 ma (5.6 V on the meter).
- (16) Illuminate the area of interest on LO. Switch to HI and complete illumination.

- (17) Set RADIATE Switch to OFF position, System Control Unit Power Switch to OFF position, and lift the MPG from the area. Check quality of patch.
- (18) After completion of the task, set Power Unit's ON-OFF switches to OFF position, Circuit Breakers to OFF position, Water Pump, Cooling Fan, and PPG switches to OFF position.
- (19) STOP PPG. (Follow instructions pertaining to the operation of PPG.)

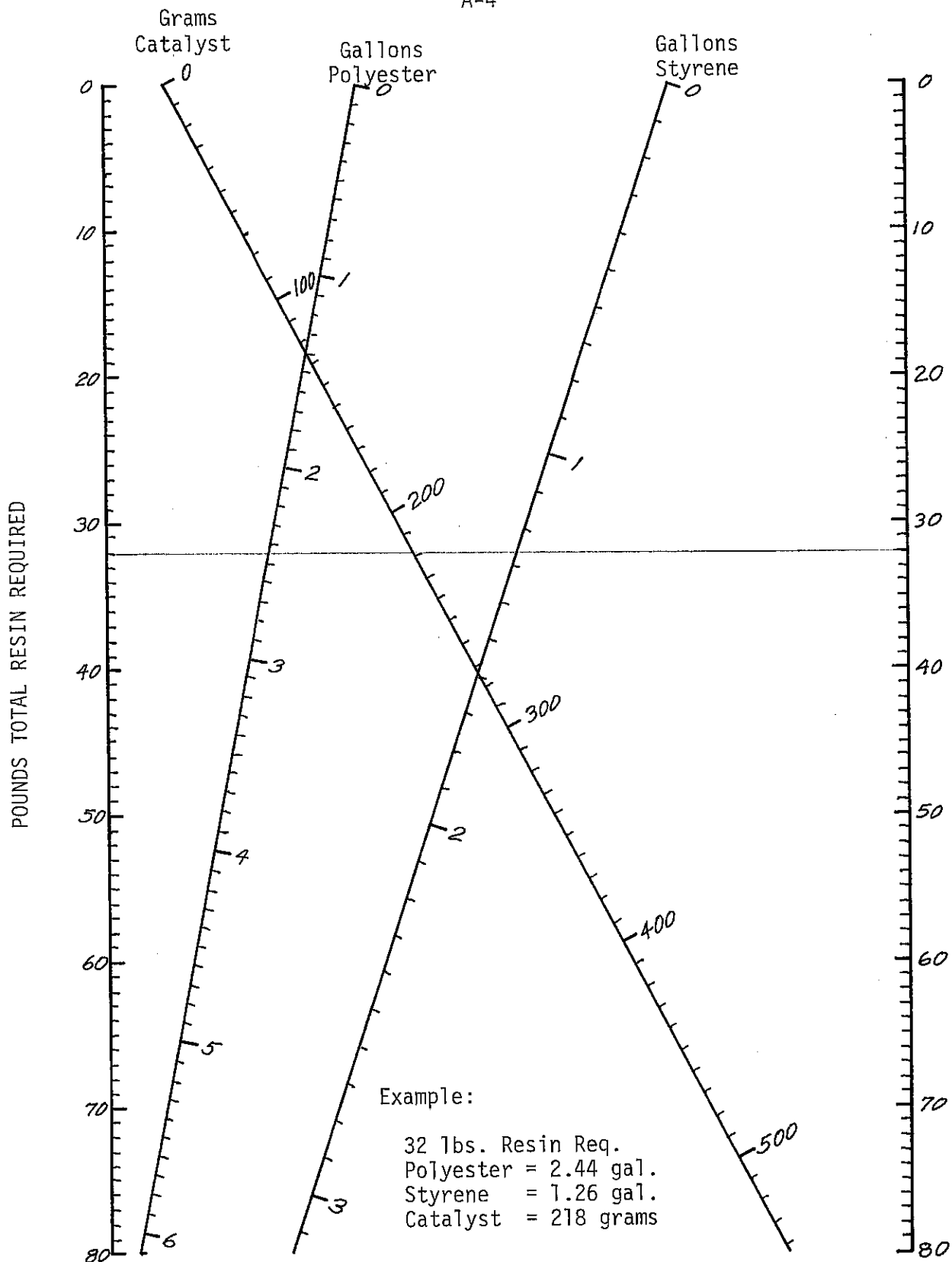


Figure A-1. Chart for proportioning 70 percent Polyester, 30 percent Styrene, and 1.5 percent Catalyst

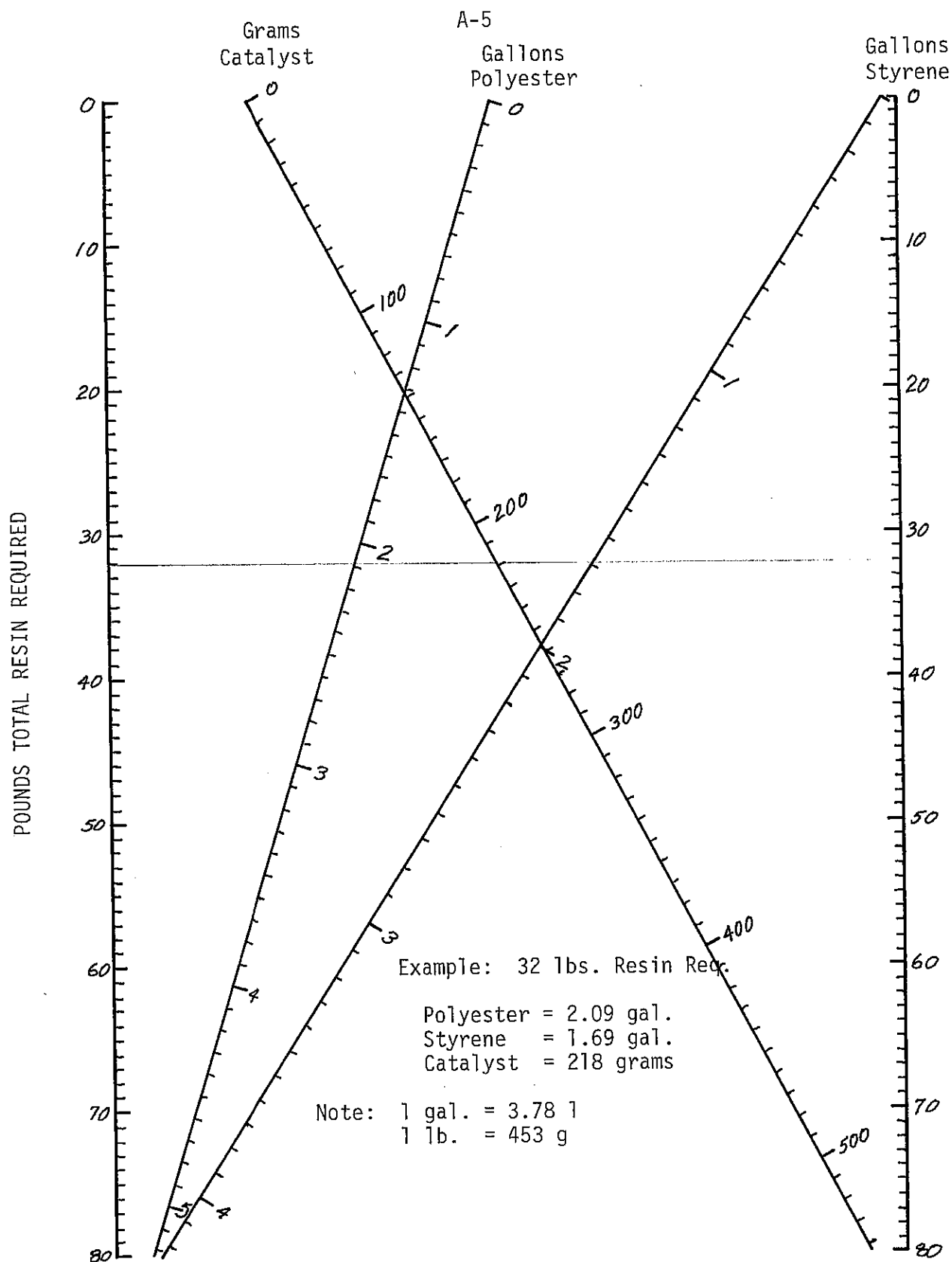


Figure A-2. Chart for proportioning 60 percent Polyester, 40 percent Styrene, and 1.5 percent Catalyst

## MATERIAL SAFETY DATA SHEET

(APPROVED BY U.S. DEPARTMENT OF LABOR "ESSENTIALLY SIMILAR" TO FORM LSB-005-4)

S-8/75

## Section I

MANUFACTURER'S NAME <b>REICHOLD CHEMICALS, INC.</b>	
STREET ADDRESS <b>525 North Broadway</b>	
CITY, STATE AND ZIP CODE <b>White Plains, New York 10603</b>	
EMERGENCY TELEPHONE NO. <b>914 682-5700</b>	
CHEMICAL NAME AND SYNONYMS <b>Unsaturated Polyester or Polyester Resin</b>	TRADE NAME <b>POLYLITE® 31-001</b>
CHEMICAL FAMILY <b>Organic Synthetic Resin</b>	FORMULA <b>Unsaturated Polyester in Monomer</b>

## Section II - HAZARDOUS INGREDIENTS

## PAINTS, PRESERVATIVES, &amp; SOLVENTS

PIGMENTS	%	TLV (Units)	SOLVENTS	%	TLV (Units)
CATALYST			ADDITIVES		
VEHICLE <b>Unsaturated Polyester</b>	<b>&gt;50</b>		OTHERS <b>Styrene Monomer</b>	<b>&lt;50</b>	<b>100ppm</b>

## HAZARDOUS MIXTURES OF OTHER LIQUIDS, SOLIDS, OR GASES

STYRENE HAZARDS: SEE MCA CHEMICAL SAFETY DATA SHEET SD-37

## Section III - PHYSICAL DATA

BOILING POINT (°F.)	Above 145°C (293°F)	SPECIFIC GRAVITY (H <sub>2</sub> O=1) (product)	1.1 - 1.2
VAPOR PRESSURE (mm Hg.) @20°C (styrene)	<5	PERCENT VOLATILE BY VOLUME (%) (product)	<50
VAPOR DENSITY (AIR=1) (styrene)	3.6	EVAPORATION RATE (n butyl acetate = 1)	<1
SOLUBILITY IN WATER (product)	N.A.		
APPEARANCE AND ODOR	Clear liquid with typical styrene odor.		

## Section IV - FIRE AND EXPLOSION HAZARD DATA

Typical Flash POINT (METHOD USED)	96°F Tag Open Cup 65°F Pensky-Martin Closed Cup	FLAMMABLE LIMITS (Vol. % in air styrene)	Lel 1.1	Uel 6.1
EXTINGUISHING MEDIA	Foam, carbon dioxide or dry chemical, per National Fire Protective Asso. Class "B" extinguisher			
SPECIAL FIRE FIGHTING PROCEDURES	None - fight like a fuel oil fire			

Unusual Fire and Explosion Hazards: Styrene will polymerize readily at elevated temperatures such as fire conditions. If this occurs in a closed container, there is a possibility of violent rupture.

THIS INFORMATION IS FURNISHED WITHOUT WARRANTY, REPRESENTATION, INDUCEMENT OR LICENSE OF ANY KIND, EXCEPT THAT IT IS ACCURATE TO THE BEST OF REICHOLD CHEMICALS, INC.'S KNOWLEDGE, OR OBTAINED FROM SOURCES BELIEVED BY REICHOLD CHEMICALS, INC. TO BE ACCURATE, AND REICHOLD CHEMICALS, INC. DOES NOT ASSUME ANY LEGAL RESPONSIBILITY FOR USE OR RELIANCE UPON SAME. CUSTOMERS ARE ENCOURAGED TO CONDUCT THEIR OWN TESTS, BEFORE USING ANY PRODUCT, READ ITS LABEL.

**Section V—HEALTH HAZARD DATA****THRESHOLD LIMIT VALUE**

Styrene 100 ppm (See Section II)

**EFFECTS OF OVEREXPOSURE**

Styrene @ 400 ppm or in strong concentration is irritating to all parts of the respiratory tract and eyes. May be fatal @ 10M ppm. Somewhat anesthetic.

(N.B.) Styrene vapor generation of polyester resins will rarely exceed 200 ppm.

**EMERGENCY AND FIRST AID PROCEDURES**

Remove victim from exposure to well-ventilated area - make comfortably warm but not hot - use oxygen or artificial respiration as required. In case of skin contact, wash thoroughly with soap and water. In case of ingestion, consult physician. In case of eye contact, flush promptly with copious amounts of water for 15 minutes and consult a physician.

**Section VI—REACTIVITY DATA****STABILITY**

UNSTABLE

**CONDITIONS TO AVOID**

Sunlight, open flames, contamination

STABLE

XX

and prolonged storage above 100°F.

**INCOMPATIBILITY (Materials to avoid)**

Strong acids, peroxides and other oxidizing agents.

**HAZARDOUS DECOMPOSITION PRODUCTS**

Carbon monoxide and dioxide, low molecular weight hydrocarbons and organic acids.

Hazardous Polymerization may occur.

Conditions to Avoid: Improper addition of promoter and/or catalyst. Consult product bulletin. A promoter (metal organic such as cobalt or aniline type) and catalyst (organic peroxide type) used with this product should always be mixed separately with the product and should never be mixed directly together. Also, sunlight, open flames, contamination and prolonged storage above 100°F.

**Section VII—SPILL OR LEAK PROCEDURES****STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED**

Remove saturated clothing promptly and wash affected skin areas with soap and water. Remove all sources of ignition (flames, hot surfaces, and electrical static, or frictional sparks). Ventilate area. Use protective measures outlined in Section VIII below. Absorb with inert materials such as Vermiculite or sand and place in closed container for disposal as solid waste. Wash area well with trisodium phosphate and water.

**WATER DISPOSAL METHOD**

Resin that may have been mixed with peroxide initiators prior to spillage should be mixed with inert filler and removed to an open area. Allow time to gel and cure. Use either approved sanitary land fill or incineration. Do not incinerate closed containers. Disposal must be carried out in accordance with local, state, and federal regulations.

**Section VIII—SPECIAL PROTECTION INFORMATION****RESPIRATORY PROTECTION (Specify type)**

Up to 100 ppm: none. 100 ppm and above: U. S. Bureau of Mines approved air line mask or self-contained breathing apparatus.

Ventilation: Provide general dilution or local exhaust ventilation to comply with Sections II and IV (styrene vapor is heavier than air). Use explosion-proof motors.

**PROTECTIVE GLOVES**

Neoprene or non-soluble plastic

**EYE PROTECTION**

Use safety eye wear designed to protect against splash or liquids.

**OTHER PROTECTIVE EQUIPMENT**

Frequently clean protective clothing, shoes, etc., to avoid skin contact with styrene. Safety showers and eye wash stations should be available.

**Section IX—SPECIAL PRECAUTIONS****PRECAUTIONS TO BE TAKEN IN HANDLING AND STORING**

Store and handle as National Fire Protective Association Class 1-C flammable liquid. Store below 27°C. (80°F.) in a closed container and dry area to avoid spoilage. Open drums slowly to relieve any internal pressure. In bulk storage check vents and flame arrestors for plugging by the formation of polymer. Ground all connections, containers, etc. when using.

This information is furnished without warranty, representation, inducement or license of any kind, except that it is accurate to the best of Reichhold Chemicals, Inc.'s knowledge, or obtained from sources believed by Reichhold Chemicals, Inc. to be accurate, and Reichhold Chemicals, Inc. does not assume any legal responsibility for use or reliance upon same. Customers are encouraged to conduct their own tests. Before using any product read its label.





# Industrial Hygiene Toxicology and Safety Data Sheet

Environmental Health Services  
Medical and Health Services Department

## Section I

Trade Name and Synonyms <b>STYRENE MONOMER</b>	
Manufacturer's Name <b>AMOCO CHEMICALS CORPORATION</b>	Emergency Telephone No. <b>(312) 856-5371</b>
Address (Number, Street, City, State and Zip Code) <b>230 EAST RANDOLPH DRIVE, CHICAGO, ILLINOIS 60601</b>	
Chemical Name and/or Family <b>STYRENE</b>	Formula <b>C<sub>6</sub>H<sub>5</sub>CH=CH<sub>2</sub></b>

## Section II Physiological Properties

Effects of Exposure	
Acute:	
Eyes	<b>CAUSES IRRITATION</b>
Skin	<b>CAUSES IRRITATION</b>
Irritation; high concentrations may have anesthetic effect.	
Respiratory System	
<b>DERMATITIS FROM REPEATED OR PROLONGED SKIN CONTACT.</b>	
Chronic:	<b>DERMATITIS FROM REPEATED OR PROLONGED SKIN CONTACT.</b>
Sensitization Properties (Species): Skin Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> Unknown <input type="checkbox"/> Respiratory Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> Unknown <input type="checkbox"/>	
Median Lethal Dose (LD <sub>50</sub> , LC <sub>50</sub> ):	Irritation Index, Estimation of Irritation (Species)
(Species)	Skin <b>MODERATE</b>
Oral <b>LD<sub>50</sub> 5.0 G/KG (RATS)</b>	Eyes <b>4 ON SCALE OF 1-10 (RABBITS)</b>
Inhalation <b>N/A</b>	Symptoms of Exposure
Dermal <b>N/A</b>	<b>N/A</b>
Other <b>N/A</b>	

## Section III Chemical and Physical Properties

Boiling Point (°F)	293.4	Vapor Pressure	4.3	Vapor Density	3.6	Specific Gravity	0.91
Melting Point (°F)	N/A	(mm Hg at 20°C)		(Air = 1)		(H <sub>2</sub> O = 1)	
pH	<b>NEUT</b>	Appearance and Odor	<b>PENETRATING, COLORLESS LIQUID</b>				
Flammable Limits % Lower	1.1	Upper	6.1	Solubility in Water	<b>NEGLECTIBLE</b>		
Products Evolved When Subjected to Heat or Combustion				<b>N/A</b>			
Hazardous Polymerization				<b>X</b> Does Not Occur			
The Material Reacts Violently With:				YES (Specify)			
Acid	<b>NO</b>	Water	<b>NO</b>	Oxygen	<b>YES</b>	Others (Specify)	
List of All Toxic and Hazardous Components (%)				List of all Nuisance Components (%)			
<b>STYRENE</b>							
This Product is Classified as:				<b>X</b>			
Hazardous (Only)							

## Section IV Control Procedures

## A. Occupational

## Protective Equipment (Type)

Eyes **CHEMICAL GOGGLES IF CONTACT IS LIKELY**Skin **GLOVES AND PROTECTIVE CLOTHING IF CONTACT IS LIKELY**Inhalation **IF VENTILATION IS INADEQUATE, USE ORGANIC VAPOR RESPIRATOR APPROVED BY MESA-NIOSH.**

## Ventilation (Type Required)

**LOCAL EXHAUST MAY BE REQUIRED**

## Precautionary Label (may be attached)

**SEE SECTION VI**Permissible Concentrations: Air **100 ppm** Biological **N/A**Monitoring Procedures Air **N/A** Biological **N/A**

## First Aid

Eyes **IMMEDIATELY FLUSH WITH WATER FOR AT LEAST 15 MINUTES. GET PROMPT MEDICAL ATTENTION.**Skin **WASH WITH SOAP AND WATER.**Ingestion **DO NOT INDUCE VOMITING; GET PROMPT MEDICAL ATTENTION.**Inhalation **Remove to uncontaminated area. Give artificial respiration if breathing has stopped. Get prompt medical attention.**

## B. Environmental

**YES**

Product is a Pollutant if discharged into:

Product is Biodegradable

Water **YES**

Not Biodegradable

Air **YES**Waste Disposal Method **CONTROLLED INCINERATION UNLESS DIRECTED OTHERWISE BY APPLICABLE ORDINANCES.**

## C. Recommended Fire Extinguishing Agents and Special Procedures

**DRY CHEMICAL (B-C), CARBON DIOXIDE, WATER FOG, FOAM**

## D. Unusual Fire and Explosive Hazards

**POLYMERIZES AT ELEVATED TEMPERATURES.****EXOTHERMIC REACTION CAN FORM EXPLOSIVE PEROXIDES ON AIR EXPOSURE.**

## E. Procedures in Case of Spillage or Leakage

**CUT OFF ALL SOURCES OF IGNITION. SOAK UP WITH AN ABSORBENT MATERIAL.**

## F. Requirements for Transportation, Handling and Storage

**STORE IN A WELL VENTILATED, FLAMMABLES STORAGE AREA AWAY FROM ALL SOURCES OF IGNITION.**

## G. NFPA Symbol



## Section V U. S. Government and Other Regulatory Agency Controls

## A. Marketing and use Regulated by (Specific Regulation)

FDA **N/A** USDA **N/A** EPA **N/A** Others (Specify) **DOT: FLAMMABLE**B. State or Local Regulations Affecting the use of this Material (Restriction on Amount Released or Discharged into Air or Water, Etc.) **N/A**

## Section VI Comments

Additional Health and Safety Information Known About The Product **WARNING! FLAMMABLE. CAUSES EYE AND SKIN IRRITATION. IRRITATING IF INHALED. Do not get in eyes, on skin or clothing. Keep away from heat, sparks and open flame. Use with adequate ventilation. Keep container closed. FOR EYES: In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Get prompt medical attention. FOR SKIN: In case of contact, wash exposed skin with soap and water. Handling Instructions: Wear chemical goggles, gloves and protective clothing if contact is likely. If clothing is contaminated, remove & launder before reuse. Wet leather shoes should be removed promptly, dried thoroughly & aired free of styrene before reuse.**

Information Supplied by:

Paul D. Halloy

Signature:

Title:

Director Environmental Health Services

Date:

September 28, 1976

N/A - Data not available.

## MATERIAL SAFETY DATA SHEET

### SECTION I

MANUFACTURER'S NAME E. I. du Pont de Nemours & Co. (Inc.)		EMERGENCY TELEPHONE NO. (302) 774-7500
ADDRESS (Number, Street, City, State, and ZIP Code) Wilmington, Delaware 19898		$(CH_3)_2C(CN)NNC(CN)(CH_3)_2$
CHEMICAL NAME AND SYNONYMS 2,2'-azobis (isobutyronitrile)		TRADE NAME AND SYNONYMS VAZO® 64 Polymerization
CHEMICAL FAMILY Azonitrile	FORMULA Initiator	$C_8H_{12}N_4$

### SECTION II HAZARDOUS INGREDIENTS OF MIXTURES

N. A. \*

### SECTION III PHYSICAL DATA

BOILING POINT (°F)	N. A.	SPECIFIC GRAVITY ( $H_2O=1$ ) (BULK)	0.28
VAPOR PRESSURE (mm Hg) low, but re- leases nitrogen on heating		PERCENT VOLATILE BY VOLUME (%)	Negligible at room temperature
VAPOR DENSITY (AIR=1)	N. A.	EVAPORATION RATE	Negligible
SOLUBILITY IN WATER	Negligible		
APPEARANCE AND ODOR White crystalline solid with no odor			

### SECTION IV FIRE AND EXPLOSION HAZARD DATA

FLASH POINT (Method used) N. A.	FLAMMABLE LIMITS gms/liter	Lel 0.02	Uel unknown
EXTINGUISHING MEDIA Carbon dioxide, dry powder, foam. Water if needed to cool other drums.			
SPECIAL FIRE FIGHTING PROCEDURES Avoid exposure to highly toxic fumes of burning or hot VAZO® 64. Wear air mask, rubber gloves and protective clothing. Fight fire from a distance.			
UNUSUAL FIRE AND EXPLOSION HAZARDS VAZO® dusts in air can explode. Avoid dusty conditions. Provide a spark-proof environment.			

### SECTION V HEALTH HAZARD DATA

THRESHOLD LIMIT VALUE Not determined
EFFECTS OF OVEREXPOSURE Exposure to dust can cause mild eye and respiratory irritation.

EMERGENCY AND FIRST AID PROCEDURES In case of skin contact with VAZO® 64 or its decomposition products, wash with plenty of soap and water; for eyes, flush with water at least 15 minutes and call a physician. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.
--

**SECTION VI REACTIVITY DATA**

STABILITY	UNSTABLE	X	CONDITIONS TO AVOID Avoid temperatures above 120°F. Decomposition may become violent.
	STABLE		

INCOMPATIBILITY (Materials to avoid)

Strong oxidizing agents such as nitric acid, inorganic peroxides.

HAZARDOUS DECOMPOSITION PRODUCTS

Tetramethylsuccinonitrile; nitrogen gas (when contained).

HAZARDOUS POLYMERIZATION	MAY OCCUR		CONDITIONS TO AVOID
	WILL NOT OCCUR	X	

**SECTION VII SPILL OR LEAK PROCEDURES**

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED

Avoid contact with eyes, skin, and clothing. Avoid breathing dust. Removesources of ignition. When decomposition does occur, do not breathe dust, vapor or decomposition products.

WASTE DISPOSAL METHOD

Do not generate dust. Can be incinerated in small quantities (less than 5 lb.)Burns vigorously. If in doubt, call Du Pont (302) 774-7500**SECTION VIII SPECIAL PROTECTION INFORMATION**

RESPIRATORY PROTECTION (Specific Type)

Use respirator mask if dusty conditions exist.

VENTILATION	LOCAL EXHAUST	SPECIAL Use air line mask for decomposition prods. creating dusty conditions.
	Maintain adequate ventilation.	
	MECHANICAL (General)	
	Unknown	

PROTECTIVE GLOVES Use rubber gloves for decomposition products.

EYE PROTECTION

Use goggles if dusty.

OTHER PROTECTIVE EQUIPMENT

None**SECTION IX SPECIAL PRECAUTIONS**

PRECAUTIONS TO BE TAKEN IN HANDLING AND STORING

Do not store in tightly sealed containers. Store in a cool, dry place, always below 75°F. Keep away from heat and direct sun.

OTHER PRECAUTIONS

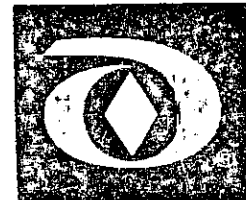
With solutions of VAZO® greater than 10% concentration, avoid exposure to heat.

\*For more information refer to: Du Pont bulletin on VAZO® 64 Product Information E-08917  
 E-05153 Du Pont bulletin "A Bibliographic Review of 2,2'-azobis  
 Rev. 8/76 (isobutyronitrile) A-65509



**Diamond Shamrock**

ELECTRO CHEMICALS DIVISION

**Material Safety  
Data Sheet**

JUL 20 1981

CHEMICAL NAME	METHYLENE CHLORIDE (Dichloromethane)	EMERGENCY PHONE NO. 216-352-7245 216-352-0022
SYNONYMS	M-Clene <sup>R</sup>	DATE FORM ISSUED
FORMULA	CH <sub>2</sub> Cl <sub>2</sub>	

**I. HAZARDOUS INGREDIENTS**

MATERIAL	TLV(UNITS)	%	MATERIAL	TLV (UNITS)	%
Methylene chloride	200 ppm	100			

**II. PHYSICAL DATA**

BOILING POINT 60 mm Hg	39.8°C (104°F)	FREEZING POINT	-96.7°C (-142°F)
SPECIFIC GRAVITY (H <sub>2</sub> O = 1)	1.32	VAPOR PRESSURE AT 20°C	352.1 mm Hg.
VAPOR DENSITY (AIR = 1)	2.93	SOLUBILITY IN WATER % BY WT. AT 20°C	Moderate
PERCENT VOLATILES BY VOLUME	100	EVAPORATION RATE (BUTYL ACETATE = 1)	Less than 1 (Ether=1)
APPEARANCE	Colorless liquid	ODOR	Ether-like

**III. FIRE AND EXPLOSION HAZARD DATA**

FLASH POINT (TEST METHOD)	None	FLAMMABLE LIMITS IN AIR (% BY VOLUME)	Upper
AUTOIGNITION TEMPERATURE	1224°F		Lower
EXTINGUISHING MEDIA	Foam, carbon dioxide, dry chemical		
SPECIAL FIREFIGHTING PROCEDURES	Self-contained respiratory protection should be provided for firemen fighting fires in buildings where methylene chloride is stored.		
UNUSUAL FIRE AND EXPLOSION HAZARDS	At high temperatures, methylene chloride can decompose giving off hydrogen chloride gas, phosgene, and other irritating vapors.		

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All information, recommendations and suggestions appearing in this literature concerning the use of our products are based upon tests and data believed to be reliable; however, it is the user's responsibility to determine the suitability for his own use of the products described herein. Since the actual use by others is beyond our control, no guarantee, expressed or implied, is made by Diamond Shamrock Corporation as to the effects of such use or the results to be obtained, nor does Diamond Shamrock Corporation assume any liability arising out of use, by others, of the products referred to herein. Nor is the information herein to be construed as absolutely complete since additional information may be necessary or desirable when particular or exceptional conditions or circumstances exist or because of applicable laws or government regulations. Nothing herein contained is to be construed as permission or as a recommendation to infringe any patent.

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## IV. HEALTH HAZARD DATA

THRESHOLD LIMIT VALUE	200 ppm by volume in air
EFFECTS OF OVEREXPOSURE	Headache, mental confusion, depression, fatigue, loss of appetite, nausea, vomiting, cough, loss of sense of balance and visual disturbances. There may also be diarrhea, suppression of urine, swelling of the face, jaundice, and blood in the urine. Severe over-exposure may cause unconsciousness and death.
EMERGENCY AND FIRST AID PROCEDURES	Remove patient immediately from the contaminated area. Obtain medical assistance as soon as possible. Wash thoroughly with water any body areas contaminated with methylene chloride.

## V. REACTIVITY DATA

STABILITY	CONDITIONS TO AVOID
<input type="checkbox"/> Unstable <input checked="" type="checkbox"/> Stable	Open flames, electrical arc
INCOMPATIBILITY (Materials To Avoid)	Aluminum, titanium, pure oxygen and alkali metals.
Hazardous Decomposition Products	Hydrogen chloride, phosgene, and other toxic products in small quantities.
Hazardous Polymerization	CONDITIONS TO AVOID
<input type="checkbox"/> May <input type="checkbox"/> Will Not Occur	

## VI. SPILL OR LEAK PROCEDURES

STEPS TO BE TAKEN IF MATERIAL IS RELEASED OR SPILLED	Spills should be cleaned up immediately. Soak up spills with rags and/or mop. When wet with methylene chloride, rags and mops should be placed in closed containers or in a safe place out of doors until they can be dried safely. Employees performing this work should wear adequate personal protective equipment. Large spills should be contained by diking. Spilled material should be picked up (vacuum truck) as soon as possible. Call emergency phone number (see front page).
WASTE DISPOSAL METHOD	Waste disposal of methylene chloride depends upon local conditions. Be sure all Federal, State, and Local regulations regarding health and pollution are followed. Methylene chloride is normally recovered from residues by distillation.

## VII. SPECIAL PROTECTION INFORMATION

RESPIRATORY PROTECTION	Self-contained breathing apparatus, positive pressure hose masks, air-line mask and industrial canister-type gas masks.		
VENTILATIONS	LOCAL EXHAUST	Satisfactory for use under normal conditions.	SPECIAL
	MECHANICAL		OTHER
PROTECTIVE GLOVES	Neoprene	EYE PROTECTION Safety goggles	OTHER PROTECTIVE EQUIPMENT

## VIII. SPECIAL PRECAUTIONS

PRECAUTIONS TO BE TAKEN IN HANDLING AND STORING	Under normal conditions, methylene chloride may be stored satisfactorily in galvanized iron, black iron or steel. Although not required, methylene chloride should be stored under pressure. Aluminum and titanium are not generally recommended for storage or handling. Store drums in a cool place, bung up and closed tightly. Ventilation should be provided at the floor level.
OTHER PRECAUTIONS	Do not store in pits, depressions and basements or in unventilated areas.